Final Report

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Ni-H_2 Cell Separator Matrix Engineering

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$Ni-H_2$ Cell Separator Matrix Engineering: Introduction to the Project and Summary of Results

This project was initiated to develop alternative separator materials to the previously used asbestos matrices which have been removed from the market for health and environmental reasons. The objective of the research was to find a material or combination of materials that had the following characteristics:

1. Resistant to the severe conditions encountered in $Ni-H_2$ cells
2. Have satisfactory electrical, electrolyte management, and thermal management properties to function properly
3. Environmentally benign
4. Capable of being manufactured into a separator matrix

During the course of the research it was discovered that separators prepared from wettable polyethylene fibers alone and in combination with potassium titanate pigment performed satisfactory in preliminary characterization tests. Further studies lead to the optimization of the separator composition and manufacturing process.

Single ply separator sheets were manufactured with 100% polyethylene fibers and also with a combination of polyethylene fibers and potassium titanate pigment (PKT) in the ratio of 60% PKT and 40% fibers. A pilot paper machine was used to produce the experimental separator material by a continuous, wet laid process. Both types of matrices were produced at several different area densities (grams/meter^2).

The PKT filled matrices exhibited more electrolyte retention than the pure polyethylene sheet and met most of the screening specifications. However, it was not possible to meet all NASA screening specifications with either of the single ply separators, but they were promising enough that both types were submitted for evaluation in pilot cells.

Further research involved the development of a new type of two ply matrix. These sheets were made by combining a 100% polyethylene sheet with a 60% PKT/ 40% fiber sheet. The two plies were bonded together by thermal calendering. Response surface experiments were carried out to determine the best manufacturing conditions. It was concluded that a satisfactory separator could be produced when a 100% polyethylene sheet (51 grams/meter^2) was bonded to a PKT filled sheet at grammages greater than or equal to 75 grams/meter^2. The respective optimum calendering conditions found from the response surface experiments were room temperature and 5 pounds per square inch. A single calender nip was found to be the best configuration.

Samples of the two ply separator matrices were produced and submitted to NASA for cell testing.

Acknowledgements

The author would like to express his sincere appreciation to NASA for sponsoring this research and to Ms. Olga Gonzalez-Sanabria and Mr. Randall Gahn, of the NASA Lewis Research Center, who served as project directors. Without their constant support and guidance, we would not have been able to carry this project to a successful completion.

Recognition should be given the many persons at Miami University who contributed to the project. Included are Dr. Albert Herbet, Mr. Richard Kling, Mr. Douglas Hart, Mr. Robin David, and Mr. Anand Kuchibhotla.

Nickel-Hydrogen Cells

Due to their potentially high capacities and low weight compared to other systems Nickel-Hydrogen batteries have been identified by National Aeronautics & Space Administration(NASA) as an important system for energy storage. In concept, the nickel-hydrogen batteries would store energy collected by solar panels during the daylight portion of a satelite's orbit and supply electrical power during the dark period of the orbit.

Nickel-Hydrogen cells are used to construct two kinds of batteries. The first type is known as an individual pressurized vessel. It is constructed by connecting several cells in series to produce the total voltage desired in the battery. Each cell contributes 1.2 volts to the total battery voltage. These batteries operate at 0-25^{o}C and 50-700 psi hydrogen pressure. The second type is known as a bipolar battery. Here, the cells are connected in parallel so that the total voltage is 1.2 volts. But the capacity of the battery is greater in this type of arrangement than in an individual pressurized vessel configuration.

The basic nickel hydrogen cell consists of a hydrogen gas anode, a nickel hydroxide cathode contained within the pores of an electrically conductive substrate and a separator that serves as an ionic bridge between the anode and the cathode. A nickel-hydrogen battery is provided with hydrogen gas storage and one or more electrode units connected electrically in parallel or series. It is called an electrode stack. In each unit a positive nickel oxide electrode is surrounded on both sides by negative hydrophilic hydrogen electrodes and separated therefrom by electrolyte saturated separators. The totality of electrodes in a common housing is designated as a

cell stack. Beyond these basic components, there is a wide variety of options for other components, depending on the methods or techniques used to assist in electrolyte management, oxygen management, and thermal management. The two main designs of nickel-hydrogen cells in current use are described below.

Back-To-Back Stack (Fig. 1)

The back-to-back stack consists of a stack of nickel- hydrogen cells and gas screens. The electrodes are connected in parallel and assembled in back-to-back configuration so that the same type of electrode faces each other. Hence, the oxygen generated during charging leaves the stack between the nickel electrodes and re-enters between the hydrogen electrodes to combine chemically with hydrogen at the catalyzed hydrogen electrode -- See Oxygen Management discussion. (Ref.3)

Recirculation Stack (Fig. 2)

The recirculation stack consists of a stack of nickel- hydrogen cells and gas screens assembled in a non back-to-back electrode configuration. The stack is packaged in a cylindrical pressure vessel with hemispherical end caps. In this type of arrangement electrodes of different types directly face each other. Hence, since a high bubble pressure separator is used, the oxygen generated by the nickel electrode during charging is directed to the hydrogen electrode of the next unit cell, where it recombines chemically to form water - -See Oxygen Management discussion.(Ref.3)

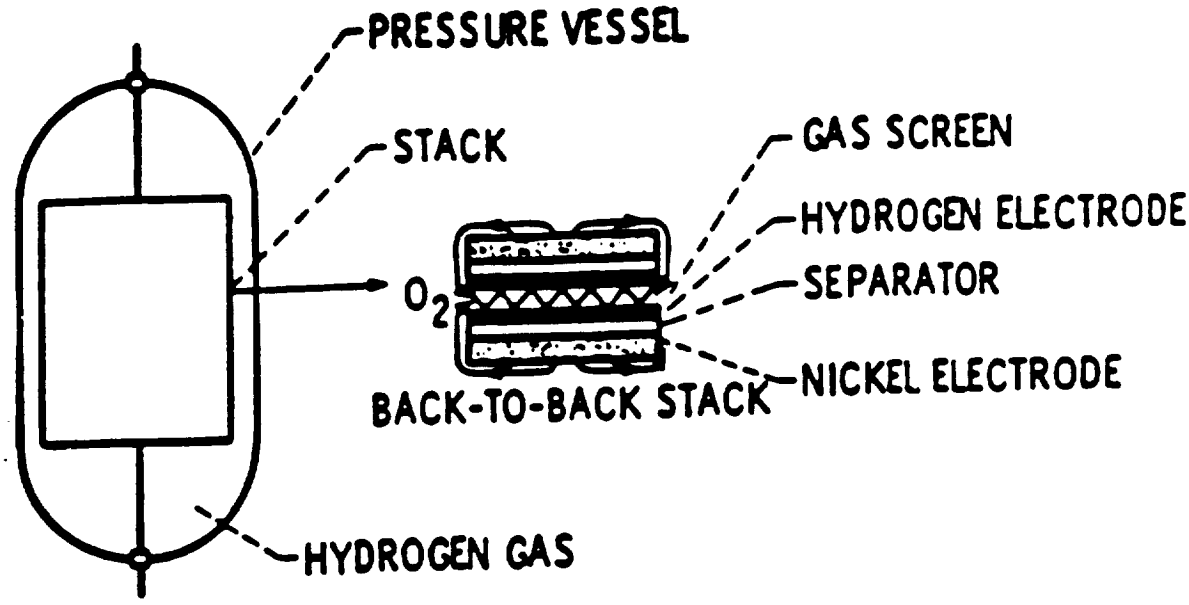


Illustration of Comsat design individual pressure vessel nickel-hydrogen cell.

Figure 1

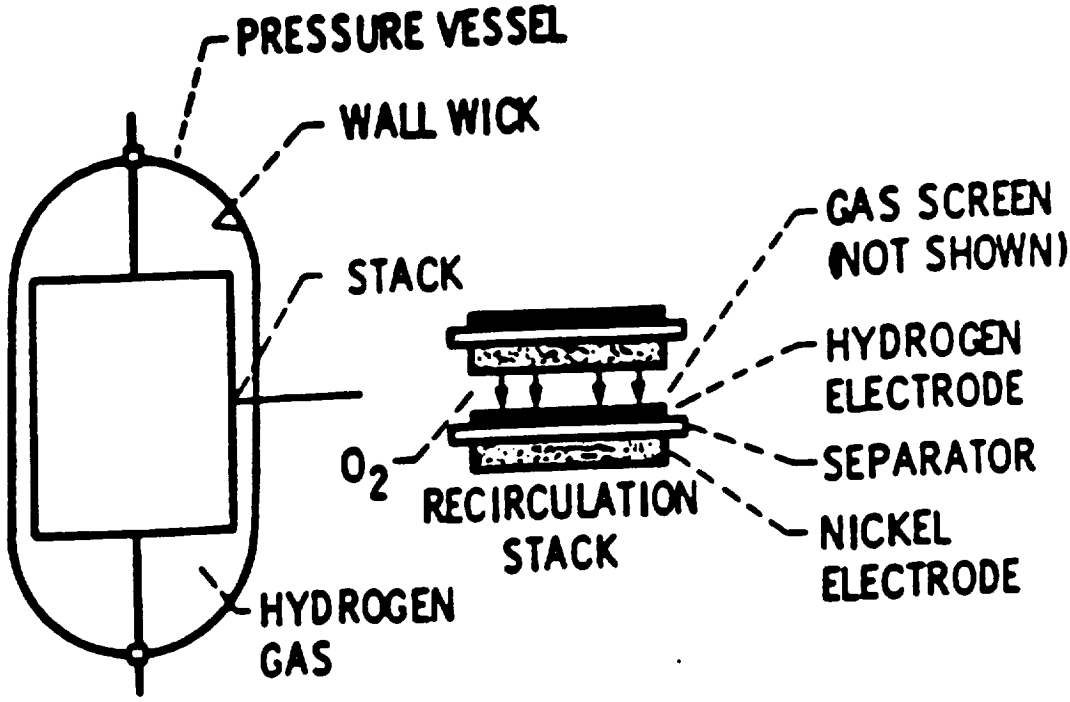


Illustration of Air Force/Hughes design individual pressure vessel nickel-hydrogen cell.

Figure 2

Cell Performance Characteristics

We will now discuss the three important cell performance characteristics, viz. Electrolyte Management, Oxygen Management, and Thermal Management.(Ref.1)

Electrolyte Management

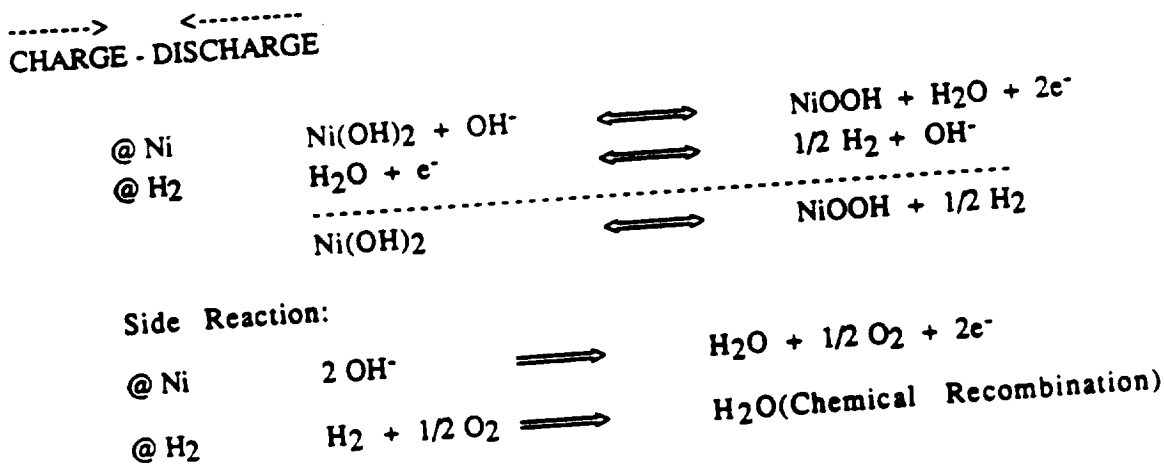
Electrolyte management can have a significant effect on the life and performance of a nickel hydrogen device and should be a prime consideration in the overall design of a cell. The objective of electrolyte management is to establish and maintain an optimum distribution of electrolyte throughout the components over the life of the device. How the electrolyte distributes itself between the components in a single cell is a very important topic and deserves utmost consideration. The combination of pore size, pore size distribution and wettability will dictate how the electrolyte is partitioned between the components as a function of the total amount of electrolyte that is added to a cell.

Nickel-Hydrogen cells are assumed to have an optimum volume. -i.e., the volume of electrolyte that will result in the highest performance. This is due mainly to the properties of the gas electrode. If the gas electrode is flooded (too much electrolyte), then the catalyst sites become blocked with electrolyte and poor performance results. If, on the other hand, the electrode is starved, (too little electrolyte) then there are not enough catalyst sites connected to the electrolyte network and again, poor performance results. What is desired is a cell design that is said to have "volume tolerance;" that is, a cell where

electrolyte can be drawn from or stored in a reservoir depending on the need. This is accomplished by the incorporation of a reservoir function into the cell separators, wherein pore size distributions are engineered such that the electrolyte reservoir will have the largest pore size, and thus the "last call" on the electrolyte. The reservoir serves to hold extra electrolyte at the beginning of cell operation for use later when either more electrolyte is required or some of the original electrolyte has been displaced for some reason.

Oxygen Management

One of the most important aspects of cell design deals with oxygen management. The nickel electrode evolves oxygen during the latter part of the charge cycle and on overcharge. This gas must be directed out of the nickel electrode and chemically recombined with hydrogen in order to maintain the proper electrolyte concentration. This is explained through the following Nickel - Hydrogen reaction equations.



In NASA developed designs , a high bubble pressure separator is used to direct the oxygen generated during the latter portions of charge and on overcharge either to an alternate catalytic surface that is between two back-to- back nickel electrodes or toward the wall of the vessel that has been treated with catalyst. This latter method provides the additional advantage of producing the heat of recombination outside the stack and on the wall of the vessel from where it is more easily removed.

As oxygen management involves water electrolysis and hydrogen and oxygen recombination, it is very closely related to electrolyte management. The wicking mechanisms and pore size distributions that serve to keep the electrolyte properly distributed also serve to redistribute the water generated from recombination.

Thermal Management

Thermal management is of particular concern because it will effect the system's performance and, ultimately, its life. The performance of the nickel electrode in regards to its discharge capacity, self discharge characteristics, and life is a function of the operating temperature. Charge acceptance by the nickel electrode is a function of temperature and state-of-charge. Thus, to maintain a set of battery cells in balance from an electrochemical point of view, only small temperature variations from cell to cell are allowable. Temperature limits must be maintained to maximize cell life. These limits are based on the desired operating temperature, electrolyte concentration, and equilibrium vapor pressure established under these conditions. When temperature and/or concentration differences exist, the vapor pressure will

seek a new equilibrium condition, forcing water vapor to transfer from place to place.

Separators

Fuel cell grade asbestos , zirconium dioxide fibres and pigmented potassium titanate are the three types of materials previously used in Ni-H_2 cell separators. None of the first two materials is optimal, however. For example, fuel cell grade asbestos separators have rather poor KOH management characteristics, undergo long-term degradation in the presence of high concentrations of KOH, and have adverse health effects.(Ref.6) The separators prepared from ZrO_2 fibers do not prevent the recombination reaction between hydrogen and oxygen from occuring, nor do they exhibit proper KOH management characteristics.

Basically, a separator acts as an ionic bridge between the electrodes.(Ref.4) A separator must resist penetration by oxygen and loose active nickel electrode material, while retaining the required chemical and thermal stability, reservoir capability, and high ionic conductivity. It is known that the separator effects the voltage characteristics of the cells and the extent of rapid oxygen recombination damage to the negative electrodes.

Separator characteristics were studied to establish those which make the separators perform effectively. The properties that affect the effective functioning of separators are resistance, electrolyte retention, electrolyte diffusion, porosity, pore size, pore size distribution and bubble pressure.

Properties of Separators

Electrolyte Diffusion

The permeability of separator matrices to the ionic constituents of the electrolyte is a contributing factor to the proper functioning of Nickel - Hydrogen cells. The permeability of the separator reflects the effect of its microstructure on the diffusion and migration of the electrolyte in a manner similar to its influence on electrical resistance properties.(Ref.2)

Pore Size

Separator pore size is a very important factor. A high pore size is favorable because of comparatively free flow of ions between the electrodes, but the individual pores must be small enough to prevent migration of the solid active materials. Also, pore sizes in the range of 2000A^{o} are important for proper electrolyte distribution in the cell.(Ref.2)

Porosity

The porosity is an important characteristic in the performance of separators. The porosity of a separator determines its ability to allow the passage of a certain amount of electrolyte. Percent electrolyte absorption is used as a measurement of porosity.

Electrolyte Retention

Electrolyte retention and absorption are important separator properties. These properties assume further significance in sealed cells with no free electrolyte present. A maximum amount of retained electrolyte is desired to

achieve minimum cell internal resistance and to maximize cell capacity over a wide range of discharge rates.(Ref.2)

Bubble Pressure

The bubble pressure test is an indication of the ability of an electrolyte saturated separator to resist the passage of gaseous oxygen bubbles.(Ref.6)

Background

Progress made in previously sponsored NASA program(Ref.7)

In the fall of 1985 the Miami University Paper Science and Engineering Department was requested by NASA to develop a research program aimed at fabricating Ni-H_2 separator matrices on the Miami pilot paper machine. The matrix composition was to be the same, or similar to a PKT/latex/ZrO_2 formulation previously developed by NASA researchers.

Accordingly, a proposal was prepared and accepted and a research program initiated. The program was concluded after two years (November, 1987) with the successful production of a separator matrix material that met the electrolyte retention and bubble pressure properties specified by NASA, but with a somewhat different composition than was originally defined. Since that time several paper machine runs have been undertaken to confirm that the material could be manufactured reproducibly and to evaluate alternative materials to the potassium titanate pigment incorporated in current research matrix. A brief chronological review of the activities carried out under the previous two grants is presented below to provide background for the proposal to be presented here.

Review of Previous Research results

Miami Experience with NASA Formulation -- The objective of the initial stage of the work at Miami was to reproduce the NASA experience with a separator matrix composed of PKT, latex, and pigmentary ZrO_2--with the difference that the Miami matrices were to be fabricated on a standard paper handsheet mold.

It was immediately apparent that it was impossible to prepare a sheet having the NASA composition on a handsheet mold. The formed "sheets" had absolutely no strength and could not be removed from the forming fabric. The reason for this was that there were no fibers present to provide strength. The formed matrix had a mud-like consistency and even after pressing and blotting, the sheet strength was too low for couching and pressing.

Experiments with ZrO_2 Tow Fibers from Zircar Inc. -- In order to increase the strength of the matrix, it was decided to replace the ZrO_2 pigment with ZrO_2 tow fibers available from Zircar, Inc. When this was done, sheets having as low as 5% ZrO_2 fibers in a PKT/latex sheet increased greatly in strength and could have as much as 20% fiber before the bubble pressure dropped below the required 20 psi. The sheets had very good strength and flexibility. Further experiments revealed that the latex levels had to be kept below 3% or the electrolyte pick-up properties of the sheet would become too low.

Paper Machine Experiments with ZrO_2 Fibers -- The next step was to attempt to make the matrix on the pilot paper machine. This was unsuccessful, however, because at the 5-20% ZrO_2 tow fiber level the sheet did not have sufficient

strength to be conveyed from the forming section to the pressing section of the paper machine. Even at the 50% fiber level, the sheet was too weak to pass through the dryers without breaking. It was clear that there was very little bonding between the ZrO_2 fibers in the wet or dry webs. In addition, many of the ZrO_2 fibers appeared to be extensively damaged by the stock preparation pumps. From these experiences, it was concluded that it would be impossible to produce satisfactory separator paper with ZrO_2 fibers on the Miami University paper machine.

Preliminary Experiments with Polyethylene Fibers -- It was decided to try wettable polyethylene fibers because other research had shown them to produce strong sheets at low weights on the Miami paper machine. Pulpex EA and Pulpex ED from Hercules were selected due to their resistance to KOH and their wettability. Both pulps have many applications in the nonwovens industry. Handsheets containing polyethylene and PKT had bubble pressure properties similar to the ZrO_2 tow fiber sheets. As much as 20% of the poly fibers could be incorporated in a sheet before the bubble pressure fell below 20 psi. In addition, the handsheets had good toughness and could be readily handled.

Paper machine experiments were undertaken and it was discovered that at 20% fiber the sheet had good strength and runnability. Its bubble pressure was lower than required, however. The results were encouraging enough to warrant further investigations.

Further Experiments with Pulpex Fibers -- Further experiments determined that hot calendering the polyethylene paper to soften and compress the fibers

increased the sheet bubble pressure to the desired levels and did not lower the electrolyte uptake performance of the material significantly. Later, it was discovered that if two press nips were run on the paper machine then the bubble pressure of the produced paper was greater than 20 psi and there was no need for hot calendering.

In addition to the PKT/polyethylene paper, a 100% polyethylene sheet was manufactured. It had essentially 0 psi bubble pressure immediately off the machine, but with calendering, the value could be increased to above 20 psi. Calendering reduced the electrolyte uptake to below the desired 100% (by weight), however.

This concluded the research carried out under the two NASA grants. Further work was undertaken on a "run-by-run" basis.

Statement of Research Problem

It is known that the separator affects the voltage characteristics of Nickel-Hydrogen cells and the extent of rapid oxygen recombination damage experienced by the negative electrodes. Therefore, it is necessary to take a closer look at the present separators, their properties and problem areas to determine the necessary characteristics for new and improved separators.

In our NASA research program the general objective is to develop a separator that is resistant to penetration by oxygen and loose active nickel electrode material, while retaining the required chemical and thermal stability, reservoir capability and high ionic conductivity.

The specific objective of the research reported here was to carry on from the previous work done here at Miami to evaluate the effects of different compositions and calendering conditions on the properties of single ply separators and two ply separators. And then to optimize such compositions and calendering conditions to produce separators that meet NASA specifications.

NASA Specifications

NASA has determined that the characteristics associated with the effective short term performance of separators are electrical resistivity, electrolyte retention, electrolyte diffusion, porosity, and bubble pressure. The following "standard" specifications were established by NASA for screening potential separator materials:

Table - I

NASA Specifications

Basis Weight	- 250 gsm
Bubble Pressure	≥ 20 psi
Electrolyte Retention	≥ 100%(by weight)
Oxidation Resistance	≤ 5% weight loss
Area Resistivity	≤ 0.7 Ohm Cm^2

EXPERIMENTAL PLAN AND PROCEDURES

EXPERIMENTAL PLAN

Experimental Program Objectives:

1. To produce a satisfactory separator where NASA specification bubble pressure and electrolyte management properties are met within the same separator. (single-ply separators.)

2. To develop a two-ply separator system where one ply controls the bubble pressure of the system and the other ply provides most of its electrolyte management characteristics. (Two-ply separators)

3. To produce a PKT-based separator matrix that will retain its integrity under Nickel-Hydrogen cell operating temperatures and KOH concentration.

4. To reduce the thickness of the matrix as much as possible without degrading the performance of the separator in the cell.

The total experimental work was mainly divided into two parts, viz. single-ply separators and two-ply separators. In both cases calendering played a vital role. The influence of different calender pressures, calendering temperatures and numbers of nips was studied on a particular separator sample with a given basis weight and composition. The results were tabulated. In the case of two-ply separators, contour plots were prepared with calender pressure on the x-axis and calender temperature on the y-axis for each of the

dependent variables, viz. bubble pressure, resistivity, electrolyte retention and thickness. Limiting boundaries were drawn on each plot corresponding to NASA specifications. Then, by using tracing paper all four optimum regions were transferred onto one plot. Any point lying in the common area of these four overlapping areas was acceptable and the area was considered as the feasible operating zone. Also, a linear multiple regression model was generated for each sample. Three dimensional response surfaces were then plotted.

In the following sections each of the objectives and experiments will be discussed. Where appropriate, supporting data and figures are included.

EXPERIMENTAL PROCEDURES

Procedure to Produce 100% Polyethylene Paper:

The formation of a uniform sheet of paper using polyethylene fibers is not a simple task, especially on a small pilot paper machine. The Miami paper machine does not have sufficient turbulence in the forming section of the machine to provide a deflocculating effect during sheet formation. Such a deflocculating action is necessary, however, if a well formed sheet is to be produced. To achieve this objective on the Miami machine, two approaches were followed. First, chemical additives were employed to aid the dispersion of the polyethylene fibers. Second, direct mechanical action was applied to the fiber slurry suspension on the forming fabric. A summary of the stock preparation and machine run conditions is presented below.

To prepare a good 100% polyethylene sheet, the polyethylene fibers must be dispersed well. Pulpex EA(50%) and Pulpex ED(50%) were mixed to make a batch of 8000 grams. Deionized water was added to make a slurry of 3.5% consistency in the beater. Then, it was circulated in the beater with no load for 60 minutes. 1500 OD grams of polyethylene fibers was taken and added to the chest. The slurry was diluted with deionized water to 200 liters. 17.5 grams of Accurac 137-high molecular weight(anionic polyacrylamide) was added while the slurry was being stirred. After this addition, the stirring was continued for 5-10 minutes. The slurry was diluted to 500 liters. An additional 17.5 grams of Accurac 137 was added and the slurry was kept under high agitation for 5-10 minutes. 3% of PVA(polyvinyl alcohol) fibre was added and stirred for 10 minutes. Deionized water was added as dilution water at the rate of 14 liters/minute.

The formed sheet was run through one press with approximately 30 psi load. The dryers were kept low enough to prevent melting of polyethylene. A positive displacement pump was used to pump the stock. The speed of the paper machine was maintained at 3.2 ft/min. The dandy roll was set in the down position with 15 psi pressure.

Procedure to Prepare PKT - Polyethylene Separator Paper:

200 grams OD (50% Pulpex EA and 50% Pulpex ED) of polyethylene was taken. 1500 gram batches were dispersed at 3.5% consistency in Waring Blender at medium speed for 1-2 minutes. 20 grams of Accurac 137 was added and stirred for 5-10 minutes. Deionized water should be used for all component preparation operations.

PKT is self dispersing, thus it was sufficient to mix this component with deionized water under normal mixing conditions. Constant agitation was maintained to avoid settling of the material to the bottom of the container. 3000 grams of PKT was added to 40 liters of water to make a PKT slurry. It was kept under agitation for 5-10 minutes. The slurry was stirred sufficiently to keep the pigment particles suspended. Then, it was diluted to 500 liters. 20 grams of $CaCl_2$ were added and stirred for 5 minutes. 3% (150 grams) of PVA fiber was added and stirred for 10 minutes. 400 ml of HCl/40 liters was added and the furnish was pumped at 300 ml /minute into the top of headbox. 10 grams of Accurac 137/40 liters was added and stirred.

The stock was pumped at 400 mls/minute to add halfway down the tray. Dilution water was added at 12-14 liters/minute. The pump used to pump the stock is a positive displacement pump. It is run through 2 press nips with 50 lbs. load.

Table - II

Paper Machine Speed

Basis Wt.	Machine Speed
62 gsm	5.9 ft/min
77 gsm	4,7 ft/min
100 gsm	3.6 ft/min

Special care was given to the paper moisture content after the drier section. To avoid cracks when the paper was wound it was better to have some moisture present.

Bubble Pressure Testing

Three samples from each separator material are cut 5 in. X 5 in. and soaked overnight in water. The sample is then placed between the two plates of the bubble pressure tester. The bubble pressure tester is then placed between two flat plates where the bottom one is supported by a hydraulic jack and the top one is fixed. The tube from the pressure gauge is connected to an air supply. The hydraulic jack is pumped until the clamping pressure is 1000 psi. The top reservoir is filled with water. The air valve is then opened slowly until the first stream of bubbles is noticed and the pressure at this point is recorded. Three samples are tested for each kind of separator paper and the

average is taken. (Bubbles may appear initially because air is entrapped between the plates. These should not be taken to be the end point.). (Picture.1)

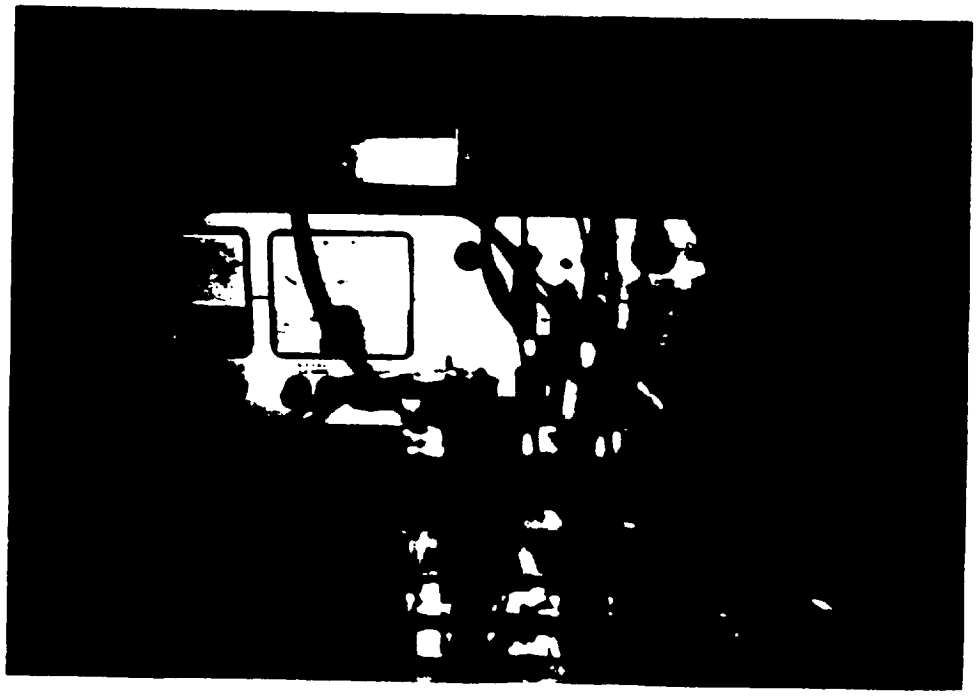
Electrical Resistivity Measurement:

A resistivity cell was constructed at Miami according to the basic design supplied by NASA. This cell is constructed of transparent plexiglass and this contains two working nickel electrodes and two zinc reference electrodes. A cell charger is required for the power supply. Two volt meters are required one - to measure the amount of current that passes through the cell and the other to measure the voltage drop across the reference electrodes due to the resistivity of the separator. (Picture 2)

Two samples from each separator material are cut to 1.5 in. x 1.5 in. .and thickness measurements are taken. They are then soaked overnight in 45% KOH at 90°C in the oven. The separator is then placed in the resistivity cell so that is is approximately centered on the cross tube that connects the two chambers. The cell is closed and two through-bolts with wing nuts tightened. 45% KOH is then allowed to flood the cell and fill the wells containing the reference electrodes up through the capillary tubes. Care is taken to prevent conduction around the cell and to exclude air bubbles from the cross tube and the capillaries.

The leads from the cell charger are then attached to the nickel electrodes. A 100 Ohms resistor is mounted in-line and the voltage drop across it measured. A 5.0 v reading on the voltmeter means that a 50 ma current is

Picture 1- Bubble Pressure Tester



Picture 2- Electrical Resistivity Tester

flowing in the cell. The voltage drop across the reference electrodes is measured on a second voltmeter. Reverse all connections and take four measurements. Then, the average is calculated. A blank is run with no separator in the cell to get the resistance of the electrolyte. This is subtracted from the resistance obtained when the separator is in the cell, the difference being the separator resistance.

Calculations

$V = IR$

Resistance of the electrolyte = $\frac{V_{blank} \text{ volts}}{0.050 \text{ amps}}$ = A Ohms

Resistance of separator = $\frac{V_{separator} \text{ volts}}{0.050 \text{ amps}}$ - A Ohms = B ohms

Area resistivity = B X area of separator exposed

Volume Resistivity = Area resistivity ÷ Thickness in Cms

Porosity Testing:

Two 5 Cm diameter circular samples are cut from each separator material. The dry weight, thickness and diameter of the specimens are measured and recorded. The samples are then soaked in 45% KOH and heated overnight at 90° C. The samples are then drained, pressed between 3 filter papers at the bottom and 2 on the top for about 30 seconds. The wet weight and thickness are then measured and recorded.

Calculation:

Porosity $\frac{W_w - W_d}{\frac{\pi}{4} \rho (L_w)(D_w^2)}$

- where W_w, W_d - wet and dry weight of separators

L_w - Thickness of wet separator

D_w - Diameter of wet separator

ρ - Density of KOH solution

The average of the two samples is calculated and then recorded.

Thickness Measurements:

Cut a separator sample of desired dimensions. Measure the thickness at 4 to 5 different places on the sample using the TMI Micrometer thickness gauge. Take the average of the readings to get the average dry thickness of the sample.

To measure the wet thickness, soak the separator in 45% KOH overnight at 90°C. The samples are then drained and pressed between filter papers. The wet thickness is measured at 5 different places on the sample to get an average wet thickness.

Electrolyte Retention

The 2 in. x 2 in. separator samples are soaked overnight in 45% KOH. The samples are then drained and pressed between filter papers to remove the excess liquid before recording the saturated properties. The separators are weighed before soaking and after soaking in electrolyte solution. The percent electrolyte retention is the ratio of final weight of the saturated separator less the initial weight over the intial weight of the dry separator.

Calculation

$$\% \text{ Electrolyte Retention} = \frac{W_w - W_d}{W_d} \times 100$$

where W_d & W_w -Dry weight & Wet weight of the sample respectively

For the ease of experimental work, water was used instead of KOH, since a direct linear correlationship was established between water retention and KOH retention and it is shown by the following equation-

KOH Pickup(Y) = 4.60 + 1.28 Water Pickup(X) $R^2 = 0.992$

Calendering of the Separator Paper

Calendering of the sheet is very crucial to achieving the required bubble pressure. The calendering operation smoothens the sheets, thus improving the bonding. The densification of the paper due to calendering also improves properties related to the handling of the paper. The sheet becomes less brittle, stronger and more resistant to handling abrasion. It is observed that starting samples having very low bubble pressures even reach values around 20 psi after calendering.

Calendering of Single Ply Separators

Pure polyethylene sheets were made at three different basis weights, while 60% PKT/40% PE sheets were made at 4 different basis weights. A 70% PKT/30% PE sheet was made at one basis weight. Several 12 in x 18 in samples were cut from the rolls directly off the machine. They were labelled according to their composition and basis weight. The following procedure was used to calender the single ply sheets.

Procedure : The calender was adjusted to 15 psi pressure. Three sheets of each separator material were then calendered at room temperature. In the same fashion, the sheets were calendered at 150°F and 170°F. At a given temperature and pressure of the calender, sheets were allowed to pass through one nip or two nips.

Preparation of Two-Ply Separators

100% PE paper was made on the machine at two different basis weights and cut into sheets of 12 in. x 18 in.. 60% PKT / 40% PE sheets were made at three different basis weights and cut into sheets of 12 in. x 18 in.. Then, one sheet of pure polyethylene and one sheet of 60% PKT/ 40% PE were put together. Altogether we obtained six different combinations. The pressure on the calender was varied from 5 psi to 20 psi in 5 psi increments. The two plies were calendered at three different levels of temperature viz., room temperature, 125°F and 135°F. The other variable was the number of nips. When passing through a second nip, the sheet was sent in the opposite direction of the first nip passing to produce a more uniform sheet. This

treatment resulted in excellent bonding between the calendered two plies. They were bonded so well that it was difficult to separate the sheets from one another.

Raw Materials

1. Polyethylene Fibres

Supplier - Hercules Inc.

Table - III

Polyethylene Properties

Grades	Pulpex EA	Pulpex ED
Length	0.8 - 1.2 mm	0.8 - 1.2 mm
Diameter	5 - 10 microns	15 - 20 microns
Melting point	130 - 135°C	130 - 135°C

2. Potassium Titanate Fiber (PKT)

Supplier - Biddle Sawyer Corporation

Brand name - TISMO: chemical name - Potassium Titanate

PKT Characteristics:

Average Length	10 - 20 microns
Diameter	0.1 - 0.3 microns

3. a.) ACCURAC 130

Supplier - American Cynamide Corporation

Characteristics - Anionnic polyacrylamide in water-in-oil emulsion

b.)ACCURAC 137

Characterstics - Cationic polyacrylamide in water-in-oil emulsion

RESULTS - DISCUSSION

This research was undertaken with the objective of producing nickel-hydrogen cell separator paper on a pilot paper machine. The papermaking mixture investigated contained various combinations of polyethylene fibers and potassium titanate (PKT).

Bubble pressure has an inverse relationship with electrolyte retention. These two specifications proved to be the most difficult to optimize in the same separator and the greatest effort was directed towards that end. The experimental program was divided into two phases:

1. Single-ply separator sheets
2 Two-ply separator sheets

The discussion of results and conclusions are presented below for both cases. Where appropriate, supporting data and figures are included.

SINGLE PLY SEPARATORS

The single ply separator sheets were manufactured with pure polyethylene fibers and also with a combination of polyethylene fibers and potassium titanate. Both types of sheets were produced at different grammages.

100% Pure Polyethylene Sheets

Grammage Vs. Separator Properties

Pure Polyethylene sheets were produced at three different basis weights:

i) 57 gsm ii) 71 gsm iii) 75 gsm. The related data is presented in Table-IV. Over this grammage range there was no correlation between grammage and bubble pressure. This is in agreement with previous results. The thickness of the separator sheet and the area resistivity were in direct relation to the weight of the sheets. But the water pickup decreased with an increase in grammage. A possible explanation for this phenomenon is that the heavier the sheet, because of the presence of more fibers and the enhanced bonding between them, the lesser the porosity (void volume) in the sheet, which has a direct influence on resistivity and water pickup.

Calendering conditions Vs. Separator Properties

The bubble pressure of the paper directly out of the machine was less than or equal to 4 psi. Cold calendering did not increase the bubble pressure more than two points, even with two nips. Once the calender was heated to 150°F and later to 170°F, there was a significant increase in bubble pressure. This is partially explained by the densification of the sheet, as can be seen in the apparent density values given in Table IV. Apparent density is the ratio of the basis weight and thickness of the sheet. The temperature chosen for the hot-calendering operation softened the polyethylene fibers, thus improving the bonding. The densification of the paper also improved properties related to the handling of the paper. The sheet became less brittle, stronger and more resistant to handling abrasion. Above 150°F the fiber tended to melt except in

the case of the 57 gsm sheet, where the bubble pressure reached a value of 21 psi at 170°F and two nips. (Table - IV)

Calendering influenced both water pickup and area resistivity tremendously, but in opposite directions. It was observed that the rate of drop in water pickup was higher at higher basis weights. The area resistivity became very high as soon as hot calendering was introduced. The measured bubble pressure - 21 psi at 170°F and two nips out of a 57 gsm sheet was the only value acceptable according to NASA specifications. As shown in Figure 3, at this bubble pressure, the water pickup was 33% and the resistivity was greater than 50 Ohms Cm^2. Neither value was acceptable.

It was concluded that it is not possible to arrive at a satisfactory separator that meets all the given specifications with a 100% pure polyethylene sheet.

60%PKT/40% Polyethylene Sheets

The separator paper with this composition was produced at four different basis weights :
i) 60 gsm ii) 70 gsm iii) 88.5 gsm iv) 134 gsm. The related data is presented in Table - V.

Grammage Vs Separator Properties

The bubble pressure directly off the paper machine was always in between 3 and 4 psi. This shows that grammage does not have any influence on bubble pressure in the uncalendered situation. With calendering, the

response was greater in sheets having higher grammage. In a similar fashion the bubble pressure values were higher for sheets with higher basis weights.

The water pickup was relatively low in uncalendered sheets when compared to pure polyethylene sheets. However, the decrease in water pickup due to calendering was not as great as for the 100% polyethylene sheet. The area resistivity decreased in the 60 gsm to 88.5 gsm range and then increased slightly at 134 gsm.

Calendering Conditions Vs Separator Properties

The bubble pressure of the uncalendered sheets was always less than or equal to 4 psi. When the sheets were calendered at room temperature, the increase in bubble pressure was usually greater than for pure polyethylene sheets.(Tables IV and V) Grammage had a clear influence on the calendering response. The increase in bubble pressure was from 3 psi for an uncalendered 60 gsm sheet to 12 psi in a sheet calendered at 170^{o}F with two nips. Whereas, the increase in bubble pressure was from 3.5 psi in an uncalendered 88.5 gsm sheet to 30 psi in a sheet calendered at 170^{o}F with two nips. The bubble pressure reached above the required 20 psi in 88.5 gsm sheets and 134 gsm sheets in the hot-calendering operation. In addition, the sheet surface became smoother and the thickness decreased implying that the calendering operation had softened the surface and improved bonding among the fibers.

There was a strong influence of calendering on both area resistivity and water pick-up. The temperature of hot-calendering influenced both area resistivity and water pickup more than the calendering pressure alone. But the decrease in water pickup was not as rapid as with pure polyethylene

sheets. The presence of PKT in the sheet influenced the water pickup. Because of their needle shape, it appears that PKT fibers create more void volume to hold water in the separator. The area resistivity values also increased with calendering but not as much as for pure polyethylene.

The bubble pressure specification was met in only two of the four sheets(89 gsm and 134 gsm), as shown in the Figures 4 and 5, where water pickup, bubble pressure and resistivity are plotted. As shown in Figure 4, at 20 psi bubble pressure, the water pickup was 67% by weight and the area resistivity was 45 Ohm Cm^2. Both of these values do not meet the NASA specifications. Even at 134 gsm and 20 psi bubble pressure , the water pickup was only 77% by weight and the area resistivity was about 42 Ohm Cm^2. Here also, both the values are out of the NASA specified range of acceptable values.

The separator paper with 70%PKT/30%PE composition was produced and tested for its properties. It showed a slight improvement in bubble pressure but not any improvement in other properties. Besides the handling of the sheet was not good enough. So it was decided not to investigate any further.

After these experiments, it was concluded that it was not possible to achieve the desired bubble pressure, area resistivity and water pickup in a single ply separator. It can also be concluded that a PKT filled polyethylene sheet has more water pickup capacity than a pure polyethylene sheet. This fact was encouraging enough to warrant further investigate into two-ply separators made out of pure polyethylene sheets and PKT filled polyethylene sheets.

Table IV

Composition, Calendering Conditions and Properties of Single Ply 100% Polyethylene Separators

One-Ply Separator Samples	Calender Pressure psi	Calender Temp. F	Bubble Pressure psi	H2O Pick-Up %	Area Resistivity Ohm Cm^2	Thickness mm	Apparent Density Kg/m^3
Type I - 57 gsm Uncalendered	-	-	3.75	219	0.04	0.30	190
1 NIP	15	RTP*	5	105	0.11	0.14	407
2 NIP	15	RTP	6	83		0.13	439
1 NIP	15	150 F	11	42	38.46	0.12	475
2 NIP	15	150 F	15	27		0.12	495
1 NIP	15	170 F	14	50	49.88	0.12	475
2 NIP	15	170 F	21	33		0.12	495
Type II - 71 gsm Uncalendered	-	-	4	210	0.18	0.33	215
1 NIP	15	RTP	6	52	0.28	0.13	546
2 NIP	15	RTP	6	33		0.12	617
1 NIP	15	150 F	10	38	40.30	0.12	617
2 NIP	15	150 F	12	22		0.10	747
1 NIP	15	170 F	13	32	51.10	0.09	789
2 NIP	15	170 F	13	13		0.09	789
Type III - 75 gsm Uncalendered	-	-	3.5	200	0.30	0.37	203
1 NIP	15	RTP	5.5	43	0.36	0 14	555
2 NIP	15	RTP	6.5	33		0.12	652
1 NIP	15	150 F	10	54	40.61	0.12	652
2 NIP	15	150 F	10	28		0.12	652
1 NIP	15	170 F	10	32	51.21	0.11	682
2 NIP	15	170 F	10	27		0.11	682

* RTP - Room Temp.

Table V

Composition, Calendering Conditions and Properties of Single Ply 60% PKT/40% PE Separators

One-Ply Separator	Calender Pressure	Calender Temp.	Bubble Pressure	H2O Pick-Up	Area Resistivity	Thickness	Apparent Density
Samples	psi	F	psi	%	Ohm Cm^2	mm	Kg/m^3
Type I - 60 gsm							
Uncalendered	-	-	3	161	0.14	0.19	316
1 NIP	15	RTP*	6	109	0.23	0.14	429
2 NIP	15	RTP	7	76		0.09	706
1 NIP	15	150 F	10	74	29.90	0.08	800
2 NIP	15	150 F	10	53		0.07	857
1 NIP	15	170 F	11	73	35.10	0.07	857
2 NIP	15	170 F	12	58		0.07	857
Type II - 70 gsm							
Uncalendered	-	-	3	152	0.04	0.22	326
1 NIP	15	RTP	8	91	0.07	0.12	609
2 NIP	15	RTP	14	67		0.10	737
1 NIP	15	150 F	15	73	30.61	0.09	778
2 NIP	15	150 F	18	63		0.08	824
1 NIP	15	170 F	17	75	37.90	0.08	824
2 NIP	15	170 F	18	60		0.08	933
Type III - 88.6 gsm							
Uncalendered	-	-	3.5	150	0.02	0.23	334
1 NIP	15	RTP	15	84	0.08	0.13	681
2 NIP	15	RTP	18	63		0.12	770
1 NIP	15	150 F	17	71	44.5	0.11	843
2 NIP	15	150 F	22	67		0.10	932
1 NIP	15	170 F	27	68	56.4	0.10	885
2 NIP	15	170 F	30	67		0.10	932
Type IV - 134 gsm							
Uncalendered	-	-	4	163	0.16	0.40	339
1 NIP	15	RTP	7	143	0.21	0.23	583
2NIP	15	RTP	14	98		0.21	654
1NIP	15	150 F	14	78	34.90	0.18	744
2NIP	15	150 F	20	77		0.17	812
1NIP	15	170 F	25	64	48.7	0.18	766
2NIP	15	170 F	29	60		0.17	812

* RTP- Room Temp.

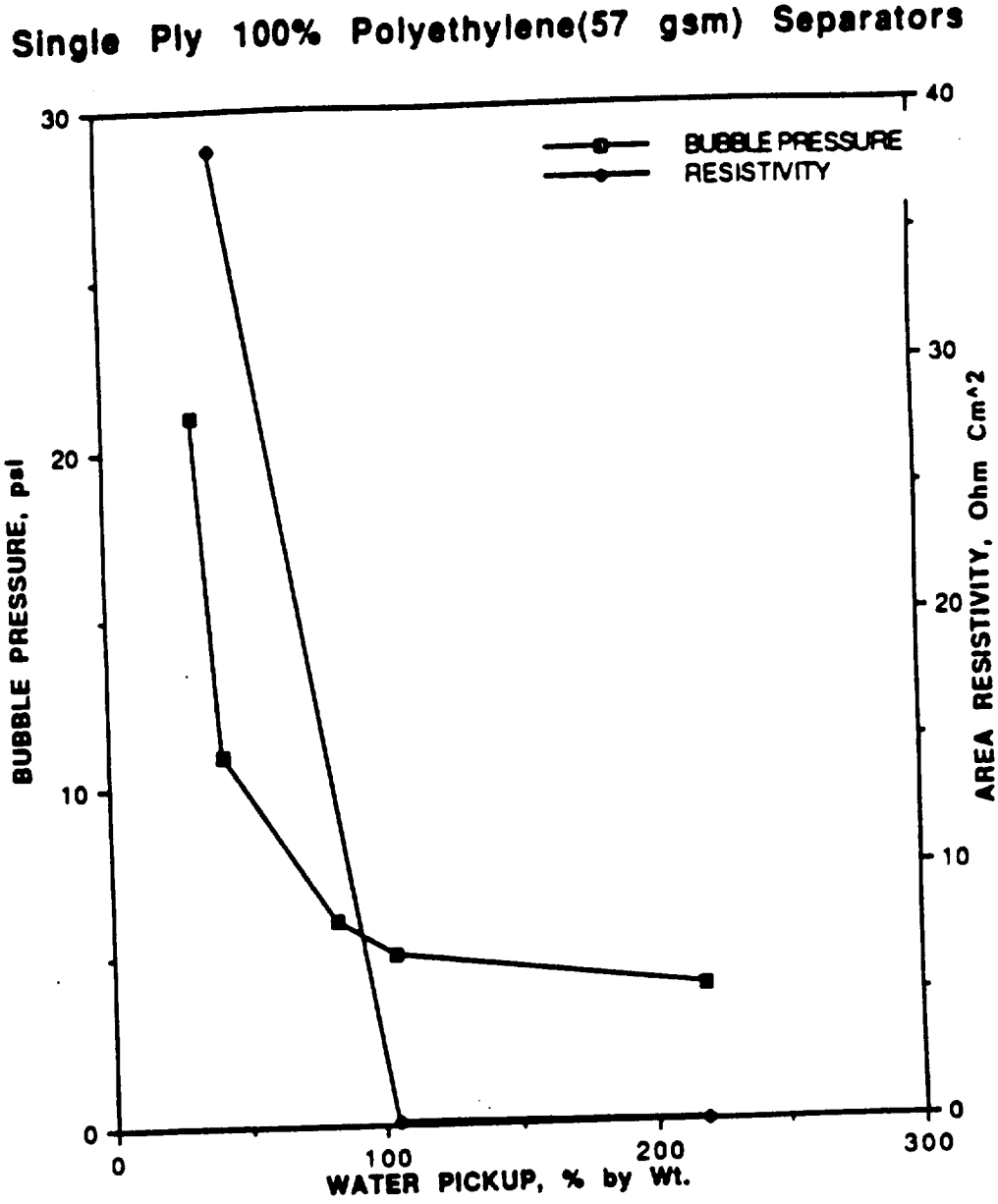


Figure 3 At 20 psi bubble pressure, water pickup is 33% by weight and resistivity is 26 Ohm Cm^2. Neither value is acceptable.

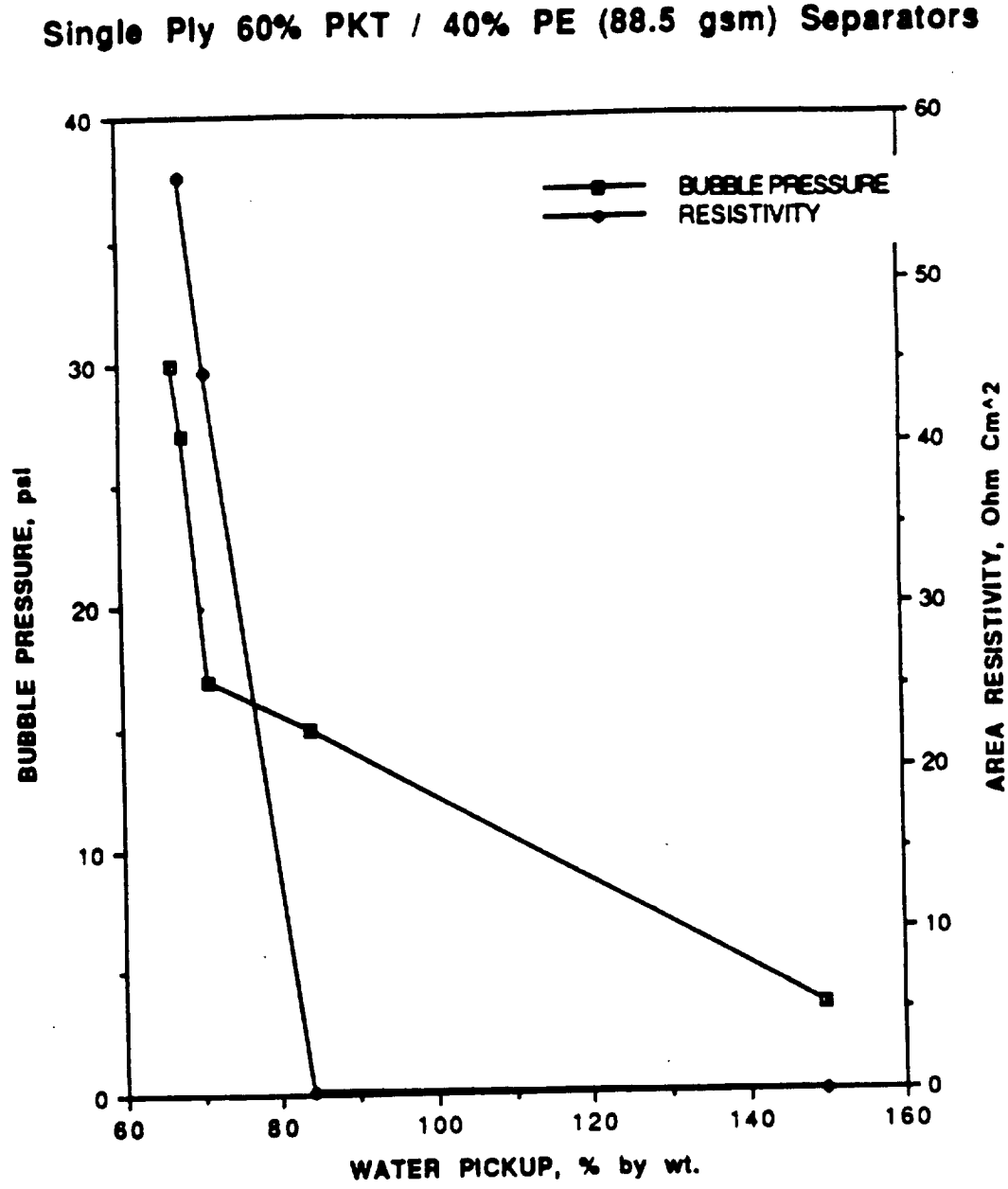


Figure 4 At 20 psi bubble pressure, water pickup is 70% by weight and resistivity is 30 Ohm Cm^2. Neither value is acceptable.

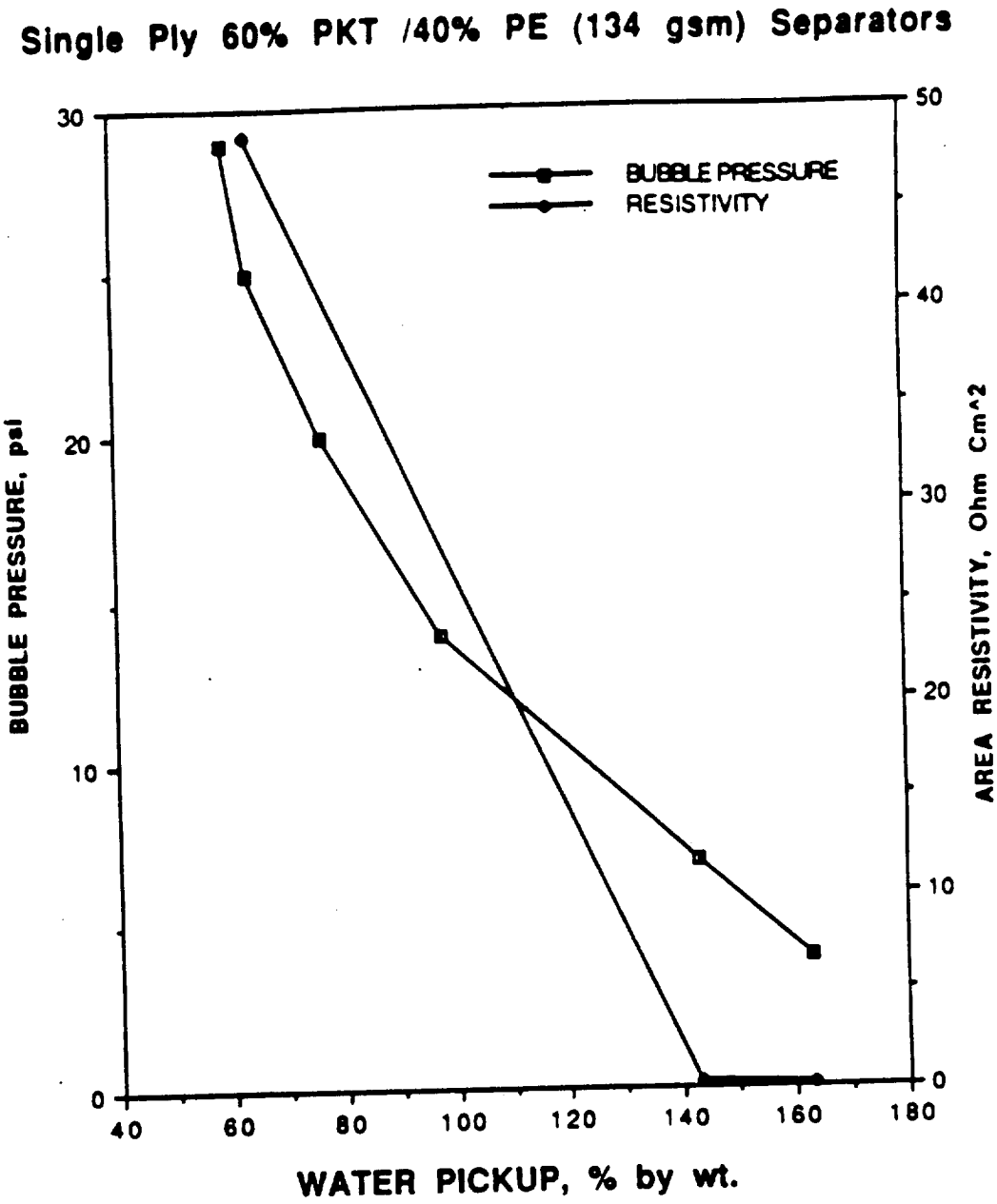


Figure 5 At 20 psi bubble pressure, water pickup is 77% by weight and resistivity is 33 Ohm Cm^2. Neither value is acceptable.

Two Ply Separators

The two ply separator sheets were made by combining a 100% polyethylene sheet with a 60%PKT/40%PE sheets. The two plies were bonded together by calendering. The 100% polyethylene sheets were made at the lowest possible basis weight (51 gsm). The 60% PKT/40% PE sheets were made at three different basis weights initially. Later on sheets at two more basis weights were produced.

Grammage Vs Separator Properties:

Since the grammage of the 100% polyethylene sheet was kept constant, the influence of grammage on separator properties was only due to changes in the 60% PKT/40% PE sheets. The related data is presented in Tables VI to VIII.

The bubble pressure of the room temperature calendered two ply separator was 9 psi when the grammage of the 60% PKT/40% PE was 63 gsm. It increased to 30+ psi when the grammage of the 60% PKT/40% PE ply was increased to 80 gsm or higher(Tables VII and VIII). (The Miami bubble pressure tester can only measure bubble pressure up-to a maximum of 30 psi.) This result showed that the grammage of the 60% PKT/40% PE sheets had a clear effect on the bubble pressure of the two ply separators.

The area resistivity of the two ply separator was 0.25 ohm Cm^2, when the 60% PKT/40%PE ply was 63 gsm. It increased to 0.64 Ohm Cm^2 when the grammage was 105 gsm. This clearly shows that the grammage of the

60%PKT/40%PE had direct influence on the area resistivity of the two ply separator.

The grammage did not have considerable influence on either water pickup or thickness. In the optimization study the 60%PKT/40%PE ply grammage was treated as a discrete, independent variable.

Calendering Conditions Vs Properties of Two Ply Separators:

Initially, the two ply separators were made from three different basis weights of 60%PKT/40%PE combined individually with a 100%PE sheet of 51 gsm basis weight. Data related to the influence of calendering conditions on the two-ply separators is presented in Tables VI - VIII. For the sake of convenience, we will refer to these three different batches as-

Batch I -(100% PE+60% PKT/40% PE(63 grams))

Batch II -(100% PE+60%PKT/40% PE (80 grams)) 87 gsm

Batch III -(100% PE+60% PKT/40%PE (105 grams))

Batch I - Two Ply Separators (See Table VI)

Calendering temperature and pressure, both had a tremendous influence on the properties of two-ply separators. In Batch I, the bubble pressure was 9 psi at 5 psi calendering pressure and room temperature. When the calendering pressure was increased to 20 psi at room temperature, the bubble pressure increased to 20 psi. When the calender was heated to 135°F at 5 psi calendering pressure, the bubble pressure exceeded 30 psi. This shows

that even though both calendering pressure and temperature had an influence on bubble pressure, the effect of temperature was the more important of the two.

Water pickup was 96% by weight at 5 psi calendering pressure and room temperature. At the same temperature and 20 psi calendering pressure, water pickup dropped to 60% by weight. The water pickup was 66% by weight at 5 psi calender pressure and 135°F temperature with two nips. This shows that water water pickup had almost the same response to both calendering temperature and pressure.

The area resistivity behaved strangely in the case of Batch I separators. Instead of increasing with increased calendering, the values exhibited no systematic variation whatsoever. No explanation could be found for this. However, the variable responded as expected in the other batches.

Batch II - Two Ply Separators (See Table VII)

Here the bubble pressure reached values exceeding 30 psi even at 5 psi calendering pressure and room temperature. The bubble pressure was above 30 psi for all the other samples also. Hence, all the samples from this batch were acceptable from this stand point.

The water pickup of Batch II separators was 82 % by weight at 5 psi calender pressure and room temperature. When the calender was heated to 135°F at 5 psi calender pressure, the water pickup dropped to 72% by weight. The water pickup had the same value at 20 psi calender pressure and room temperature. In this batch of separator samples, both high calender pressure

and high calender temperature had the same influence on the water pickup property of the separators. Furthermore, in Batch II some samples had the required 80% by weight water pickup along with the necessary bubble pressures.

The area resistivity was 0.58 Ohm Cm^2 at 5 psi calender pressure and room temperature. It increased to 1.27 Ohm Cm^2 at the same pressure but at 135°F calender temperature. When the calender pressure was increased to 20 psi, the area resistivity increased to 0.91 Ohm Cm^2. At the same calender pressure, but when the calender was heated to 135°F, the area resistivity was 1.27 Ohm Cm^2. This clearly shows that area resistivity was directly related to both calender temperature, calender pressure and the number of nips, but the response was more to calender temperature. Here a few samples had the satisfactory area resistivity values.

The calender temperature had a very minimal influence on the thickness of the separator. Except at the initial calender pressure, the response to higher calender pressures was also relatively slow.

In this batch of separator samples, a few had all the required properties. The conditions relevant to these samples were used in the subsequent optimization studies discussed below.

Batch III - Two Ply Separators (See Table VIII)

Here the bubble pressure reached values exceeding 30 psi for all samples, even at 5 psi calendering pressure and room temperature. The

bubble pressure was above 30 psi for all the other samples also. Hence, all the samples from this batch were acceptable from this stand point.

The water pickup of Batch III separators was 80% by weight at 5 psi calender pressure and room temperature. When the calender was heated to 135°F at 5 psi calender pressure, the water pickup dropped to 72% by weight. The water pickup had a value of 76% by weight at 20 psi calender pressure and room temperature. In this batch of separator samples, both high calender pressure and high calender temperature had the same influence on the water pickup property of the separators. Furthermore, in Batch II some samples had the required 80% by weight water pickup along with the necessary bubble pressures.

The area resistivity was 0.64 Ohm Cm^2 at 5 psi calender pressure and room temperature. It increased to 1.04 Ohm Cm^2 at the same pressure and 135°F calender temperature. When the room temperature calender pressure was increased to 20 psi, the area resistivity increased to 1.05 Ohm Cm^2. At the same calender pressure, but when the calender was heated to 135°F, the area resistivity was 1.27 Ohm Cm^2. This clearly shows that area resistivity was directly related to both calender temperature, calender pressure and the number of nips, but the response was more to calender temperature. Here also, a few samples had satisfactory area resistivity values.

The calender temperature had a very minimal influence on the thickness of the separator. Except at the initial calender pressure, the response to higher calender pressures was also relatively slow.

In this batch of separator samples, a few had all the required properties. The conditions relevant to these samples were used in the subsequent optimization studies discussed below.

Table VI

Calendering Conditions and Properties of Two Ply Separators Batch I: 100% PE (51 gsm) + 60% PKT/40% PE (63 gsm)

Two-Ply Separator Samples BATCH - I	Calender Pressure psi	Calender Temp. F	Bubble Pressure psi	Water Pick-Up %	Area Resistivity Ohm Cm^2	Thickness mm
1 NIP	5	RTP*	9	96	.25	0.20
2 NIP	5	RTP	12	88	.49	0.17
1 NIP	5	125 F	15	76	.33	0.17
2 NIP	5	125 F	17	66	.47	0.16
1 NIP	5	135 F	17	70	.38	0.17
2 NIP	5	135 F	19	66	.45	0.15
1 NIP	10	RTP	13	81	.47	0.17
2 NIP	10	RTP	17	68	.84	0.16
1 NIP	10	125 F	21	68	.40	0.16
2 NIP	10	125 F	27	64	.45	0.15
1 NIP	10	135 F	24	58	.35	0.15
2 NIP	10	135 F	25	51	.39	0.15
1 NIP	15	RTP	16	67	.74	0.15
2 NIP	15	RTP	19	66	1.10	0.15
1 NIP	15	125 F	25	63	.62	0.15
2 NIP	15	125 F	28	57	.70	0.15
1 NIP	15	135 F	27	52	.55	0.14
2 NIP	15	135 F	29	49	.57	0.14
1 NIP	20	RTP	20	60	1.14	0.15
2 NIP	20	RTP	22	57	1.21	0.15
1 NIP	20	125 F	27	58	.87	0.14
2 NIP	20	125 F	28	63	.87	0.14
1NIP	20	135 F	29	62	.68	0.14
2 NIP	20	135 F	30+	45	.71	0.14

* RTP – Rm Temp.

Table VII

Calendering Conditions and Properties of Two Ply Separators Batch II: 100% PE (51 gsm) + 60% PKT/40% PE (89 gsm)

Two-Ply Separator Samples BATCH - II	Calender Pressure psi	Calender Temp. F	Bubble psi	H2O Pick-Up %	Area Resistivity Ohm Cm^2	Thickness mm
1 NIP	5	RTP*	30+	82	.58	0.17
2 NIP	5	RTP	"	81	.67	0.17
1 NIP	5	125 F	"	82	.76	0.18
2 NIP	5	125 F	"	72	.89	0.17
1 NIP	5	135 F	"	79	1.12	0.17
2 NIP	5	135 F	"	72	1.27	0.17
1 NIP	10	RTP	30+	79	.65	0.17
2 NIP	10	RTP	"	79	.81	0.16
1 NIP	10	125 F	"	69	.92	0.16
2 NIP	10	125 F	"	66	1.01	0.16
1 NIP	10	135 F	"	65	1.25	0.16
2 NIP	10	135 F	"	62	1.39	0.16
1 NIP	15	RTP	30+	75	.87	0.17
2 NIP	15	RTP	"	68	.86	0.16
1 NIP	15	125 F	"	63	1.05	0.16
2 NIP	15	125 F	"	62	1.25	0.16
1 NIP	15	135 F	"	62	1.52	0.16
2 NIP	15	135 F	"	59	1.70	0.16
1 NIP	20	RTP	30+	72	.91	0.16
2 NIP	20	RTP	"	66	.99	0.16
1 NIP	20	125 F	"	61	1.35	0.16
2 NIP	20	125 F	"	58	1.54	0.15
1NIP	20	135 F	"	60	1.62	0.16
2 NIP	20	135 F	"	57	1.72	0.15

* RTP - Room Temp.

Table VIII

Calendering Conditions and Properties of Two Ply Separators Batch III: 100% PE (51 gsm) + 60% PKT/40% PE (105 gsm)

Two-Ply Separator Samples BATCH - III	Calender Pressure psi	Calender Temp. F	Bubble psi	H2O Pick-Up %	Area Resistivity Ohm Cm^2	Thickness mm
1 NIP	5	RTP*	30+	80	.64	0.20
2 NIP	5	RTP	"	82	.77	0.19
1 NIP	5	125 F	"	87	.86	0.20
2 NIP	5	125 F	"	80	.91	0.20
1 NIP	5	135 F	"	81	1.03	0.20
2 NIP	5	135 F	"	72	1.04	0.19
1 NIP	10	RTP	30+	86	.82	0.20
2 NIP	10	RTP	"	80	.89	0.19
1 NIP	10	125 F	"	77	1.01	0.19
2 NIP	10	125 F	"	70	1.28	0.18
1 NIP	10	135 F	"	69	1.34	0.18
2 NIP	10	135 F	"	66	1.39	0.18
1 NIP	15	RTP	30+	78	.88	0.20
2 NIP	15	RTP	"	74	1.07	0.19
1 NIP	15	125 F	"	69	1.21	0.19
2 NIP	15	125 F	"	65	1.33	0.18
1 NIP	15	135 F	"	65	1.47	0.18
2 NIP	15	135 F	"	58	1.24	0.17
1 NIP	20	RTP	30+	76	1.05	0.18
2 NIP	20	RTP	"	69	1.10	0.18
1 NIP	20	125 F	"	62	1.22	0.17
2 NIP	20	125 F	"	61	1.35	0.17
1NIP	20	135 F	"	63	1.53	0.17
2 NIP	20	135 F	"	60	1.66	0.16

* RTP- Room Temp

Determination of Calendering Conditions Required To Produce Acceptable Separator Material

Contour Plots

Since several different independent and dependent variables were involved in this study, it was necessary to employ some technique to arrive at the final range of operating conditions that would produce acceptable two-ply separators. This was accomplished with the aid of contour plots.

Several contour plots were generated with calendering pressure on the X-axis and calendering temperature on the Y-axis. The number of nips was treated as a discrete variable. For each batch of separators, three contour plots were plotted for each variable, viz, bubble pressure, area resistivity and water pickup. Since the number of nips was treated as a discrete variable, two sets of contour plots were generated for each batch of separators.

Batch I- Contour plots (See Figures 6 and 7)

Six contour plots were produced, as shown in Figures 6 and 7. In the bubble pressure contour plot, contour lines were generated in increments of 2 psi starting with 10 psi at the low end and going to 24 psi. The region to the right of a given contour line represents the values higher than the contour line value, showing that bubble pressure increased with increasing calender pressure and calender temperature.

In the area resistivity contour plots, contour lines were generated in increments of 0.1 Ohm Cm^2 starting at 0.3 Ohm Cm^2 at low end and going to 1.1 Ohm Cm^2. The region to the right of a given contour line represents values

greater than the contour line value, showing that area resistivity increased with increasing calender pressure and calender temperature.

In the water pickup contour plot, contour lines were generated in increments of 5% from 55% at the low end and going to 95%. The region to the right of a given contour line represents values lower than the contour line value. It shows that water pickup decreases with increasing calender temperature and calender pressure.

Batch II - Contour Plots (See Figure 8.)

Here the bubble pressure was well above the required 20 psi level for all separator samples. Therefore no contour lines could be plotted and the whole region is an acceptable region.

In the area resistivity contour plots, contour lines were plotted in increments of 0.1 Ohm Cm^2, from 0.6 Ohm Cm^2 to 1.6 Ohm Cm^2 for one nip and 0.7 Ohm Cm^2 to 1.7 Ohm Cm^2 for two nips . In both cases, the region to the right of a given contour line represents values higher than the contour line value.

In the water pickup contour plots, contour lines were plotted in increments of 5%, from 65% to 80% for one nip and from 60% to 80% for two nips . In both cases, the region to the right of a given contour line represents the values lower than the contour line value.

Batch III - Contour Plots (see Figure 9.)

Here the bubble pressure was well above the required 20 psi level for all separator samples. Therefore no contour lines could be plotted and the whole region is an acceptable region.

In the area resistivity contour plots, contour lines were plotted in increments of 0.1 Ohm Cm^2, from 0.6 Ohm Cm^2 to 1.5 Ohm Cm^2 for one nip and 0.8 Ohm Cm^2 to 1.6 Ohm Cm^2 for two nips . In both cases, the region to the right of a given contour line represents values higher than the contour line value.

In the water pickup contour plots, contour lines were plotted in increments of 5%, from 65% to 85% for one nip and from 60% to 80% for two nips . In both cases, the region to the right of a given contour line represents the values lower than the contour line value.

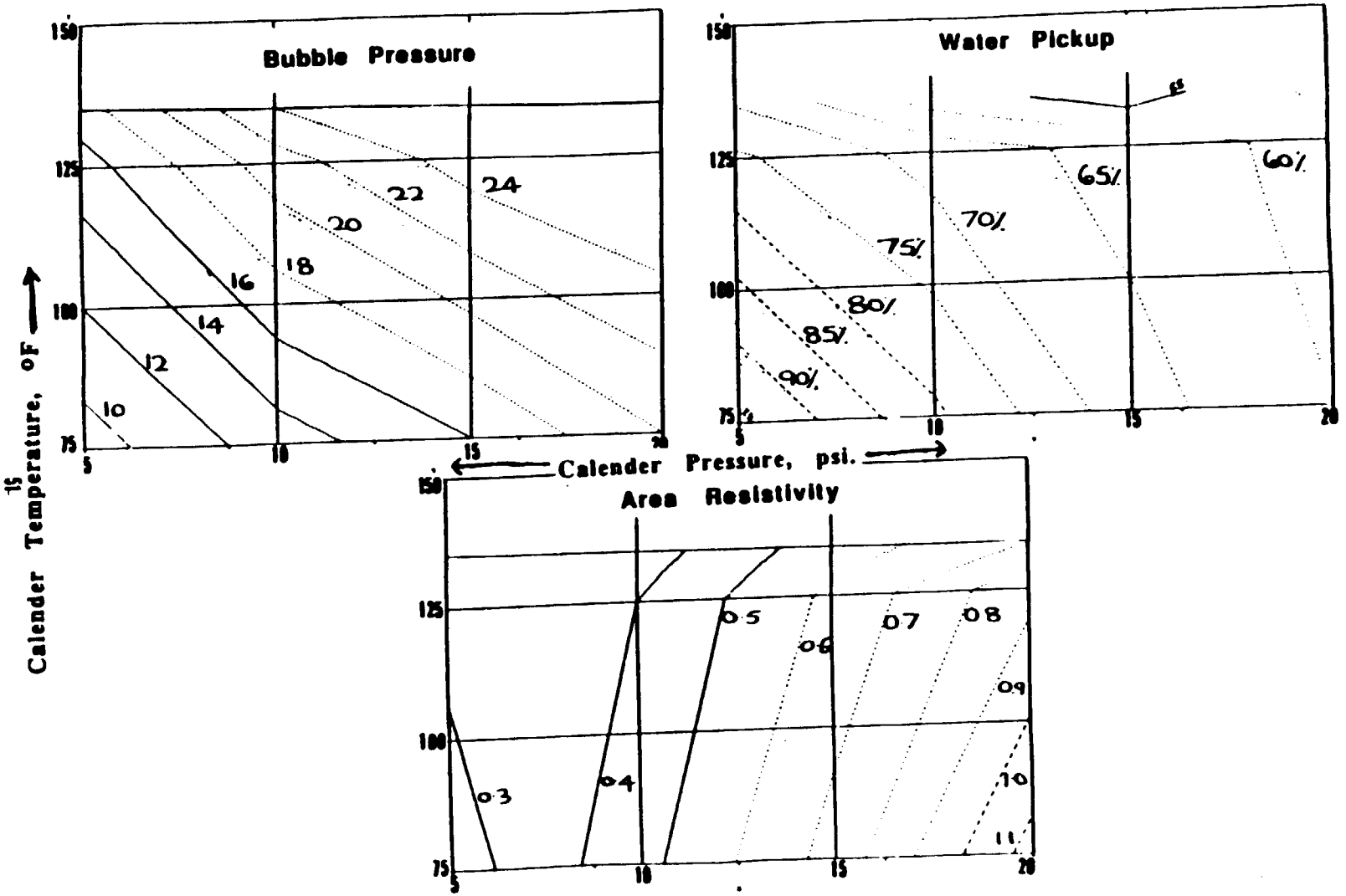


Figure 6 Contour Plots of Batch I Two Ply Separators - One Nip
Batch I: 100% PE (51 gsm) + 60% PKT/40% PE (63 gsm)

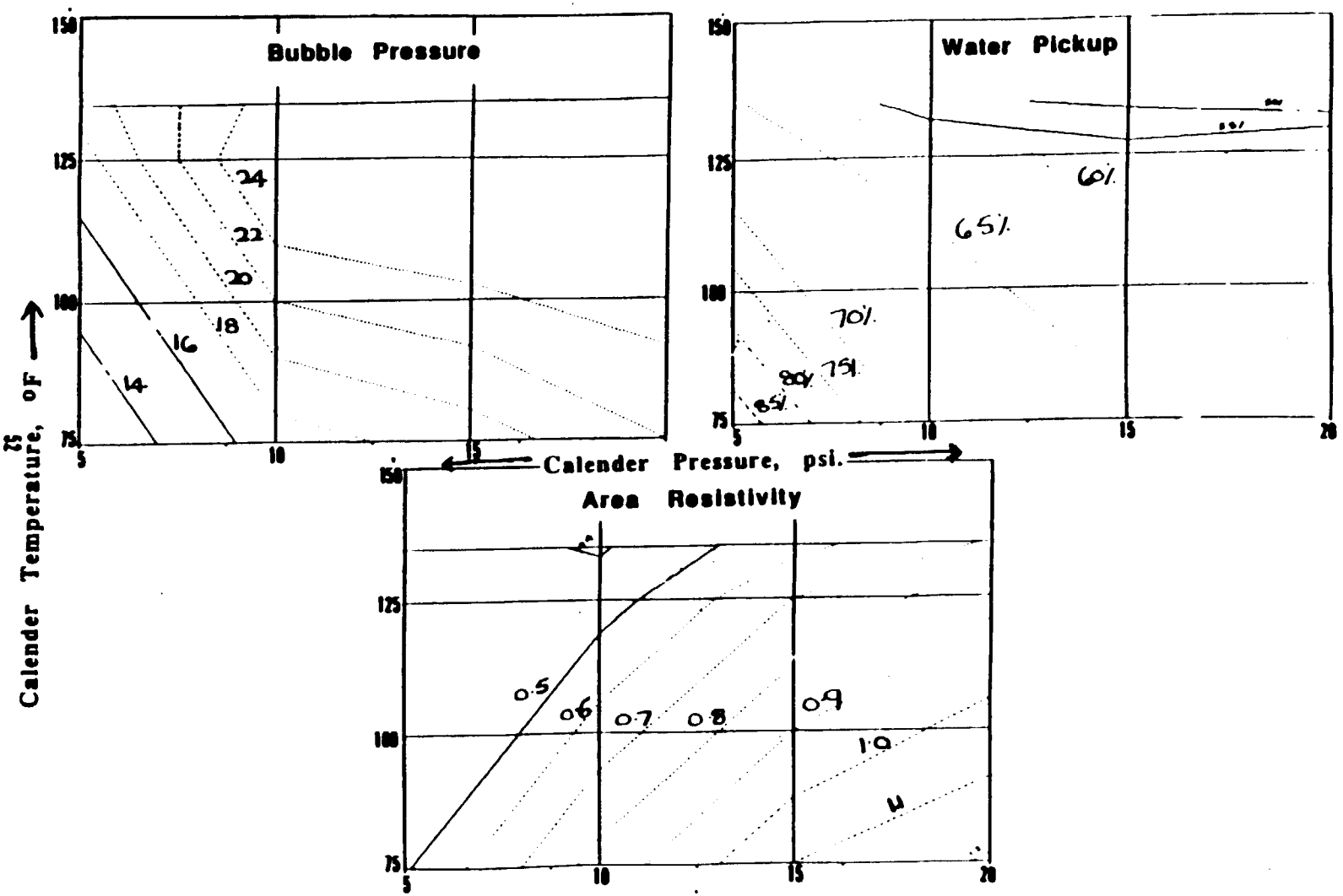


Figure 7 Contour Plots of Batch I Two Ply Separators - Two Nips
Batch I: 100% PE (51 gsm) + 60% PKT/40% PE (63 gsm)

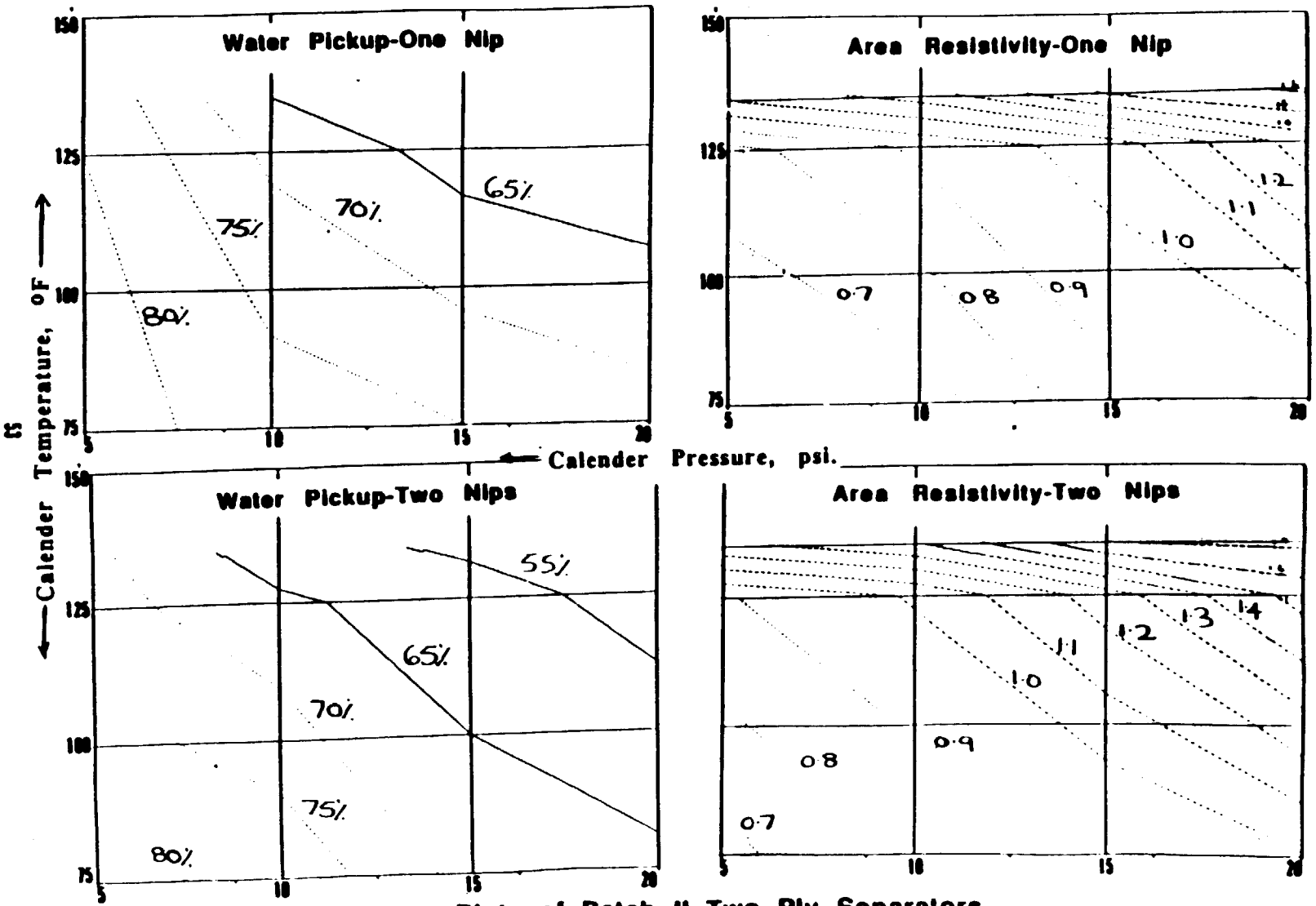


Figure 8 Contour Plots of Batch II Two Ply Separators
Batch II: 100% PE (51 gsm) + 60% PKT/40% PE (89 gsm)

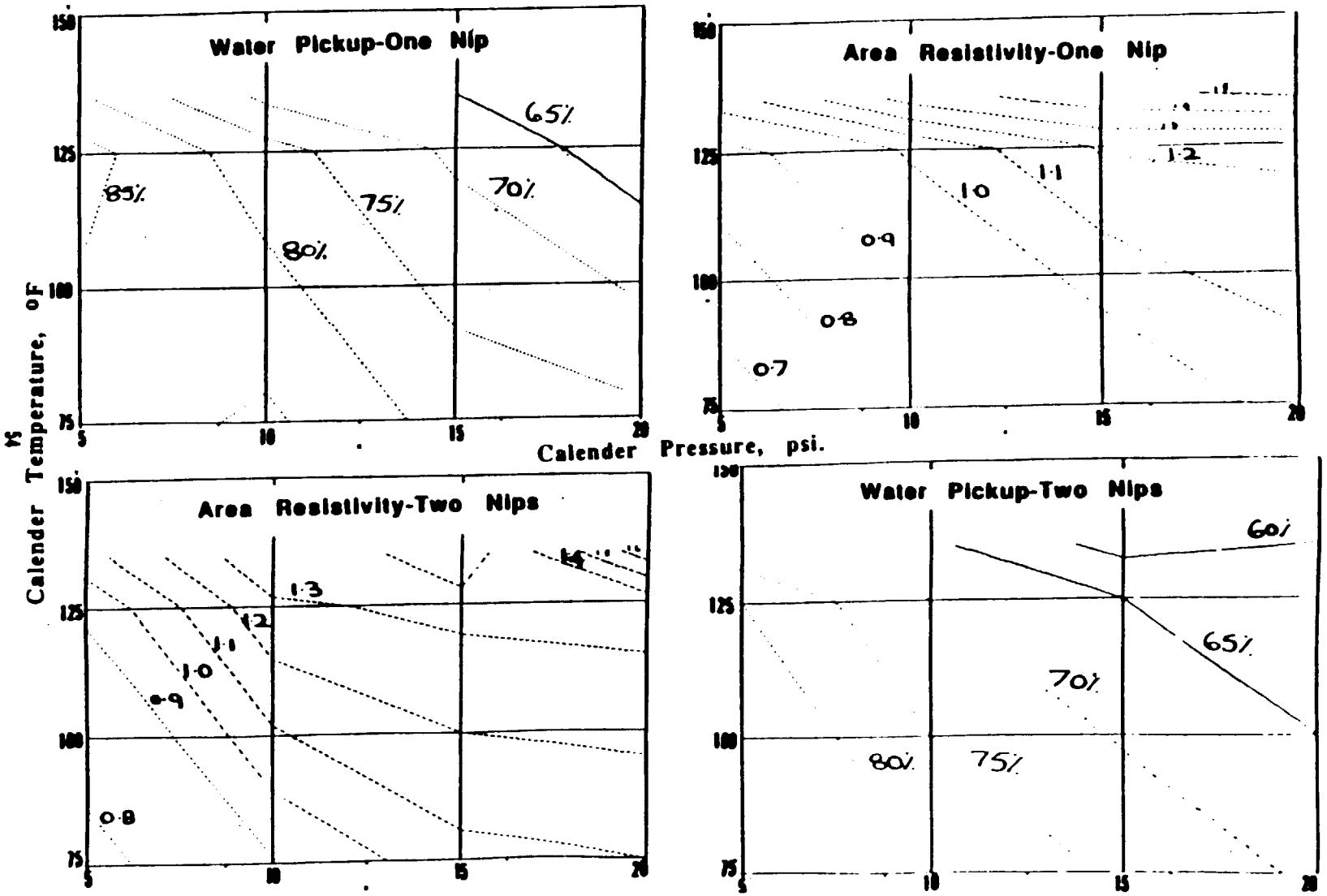


Figure 9 Contour Plots of Batch III Two Ply Separators
Batch III: 100% PE (51 gsm) + 60% PKT/40% PE (105 gsm)

Determination of Required Calendering Conditions - Batch I Separators

Batch I Separators had six contour plots - three for one nip and three for two nips. From the six contour plots, the contour lines corresponding to the separator properties, viz. bubble pressure, area resistivity and water pickup, that met the NASA specifications were transfered to one plot using tracing paper. (Figure 10)

The areas to the left of the water pickup and area resistivity lines consist of all those points that meet the NASA specifications. Any point lying in these areas was acceptable. Likewise the area to the right of the bubble pressure line consists all those points that are acceptable according to NASA specifications. To obtain the optimum composition and calendering conditions to arrive at a satisfactory separator that has the required bubble pressure, area resistivity and water pickup, there should be some overlapping or common region among these three areas. For Batch I separators, there was no such common area. Therefore, under these calendering conditions, it was not possible to produce a separator that simultaneously meets the standards for the three different properties.. Thus, it was concluded that we could not get a satisfactory separator out of the Batch I formulations and conditions.

Determination of Required Calendering Conditions - Batch II Separators

Since the bubble pressure was above 30 psi for all the separators in this batch, the entire plot was acceptable. Batch II Separators had four contour

plots - two for one nip and two for two nips. From the four water pick-up and area resistivity contour plots, the contour lines corresponding to the area resistivity and water pickup regions that met NASA specifications were transfered to one plot. (Figure 11)

The areas to the left of the water pickup and area resistivity lines consist of all points that meet the NASA specifications. Any point lying in these areas is acceptable. Bubble pressure is acceptable over the entire region. All calendering conditions that will produce separators having the required bubble pressure, area resistivity and water pickup lie in the common region among these three areas. In this plot the shaded area with horizontal lines was common to the three different properties for papers receiving one nip calendering corresponding conditions to any point in the shaded area would produce a satisfactory separator. The shaded area with the vertical lines was obtained as a common zone when the sheets were calendered with two nips. It is concluded that a satisfactory separator can be produced out of this batch of separators at 5 psi calender pressure and room temperature

Determination of Required Calendering Conditions - Batch III Separators

Since the bubble pressure was above 30 psi for all the separators Batch III the entire plot was acceptable. Batch III Separators had four contour plots - two for one nip and two for two nips. From the four contour plots, the contour lines corresponding to the separator properties, viz. area resistivity and water pickup regions that met NASA specifications were transfered to one plot. (Figure 12)

The areas to the left of the water pickup and area resistivity lines consist of all points that meet the NASA specifications. Any point lying in these areas is acceptable. Bubble pressure is acceptable over the entire region. All calendering conditions that will produce separators having the required bubble pressure, area resistivity and water pickup, there should be some overlapping or common region among these three areas. In this plot the shaded area with horizontal lines was common to the three different properties. It means the calendering conditions that gave rise to any point in the shaded area were good enough to produce a satisfactory separator. There was no common zone, when the sheets were calendered with two nips. It is concluded that a satisfactory separator could be produced out of this batch of separators at 5 psi calender pressure, room temperature and the sheets calendered with one nip.

After the initial three batches, two more batches of separators were produced with the following composition.

Batch II/1 - 100%PE (51 gsm) + 60%PKT/40%PE (69 gsm)

Batch II/2 - 100%PE (51 gsm) + 60%PKT/40%PE (75 gsm)

These two batches of separators were also tested for different properties of separators.(Table IX) After these testing, it was concluded that to produce a satisfactory separator a 100%PE(51 gsm) sheet should be combined with a 60%PKT/40%PE sheet with a basis weight of 75 gsm. The relationship between the basis weight of 60%PKT/40%PE component of the two ply separator and separator properties is shown in Figure 13. It can be clearly seen that the basis weight had a clear influence on separator properties.

Determination of Required Calendering Conditions
Batch I: 100% PE (51 gsm) + 60% PKT/40% PE (63 gsm)

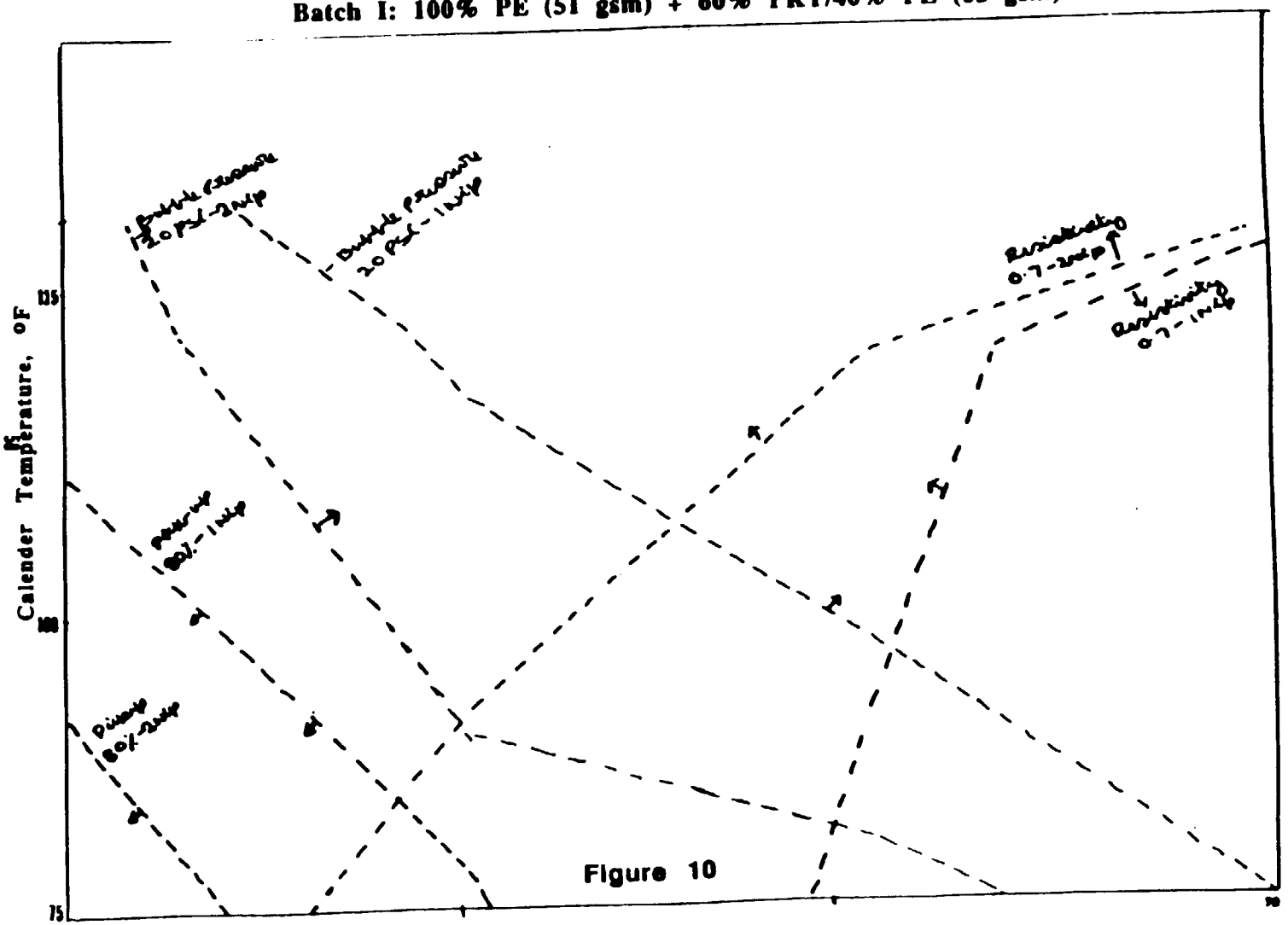


Figure 10

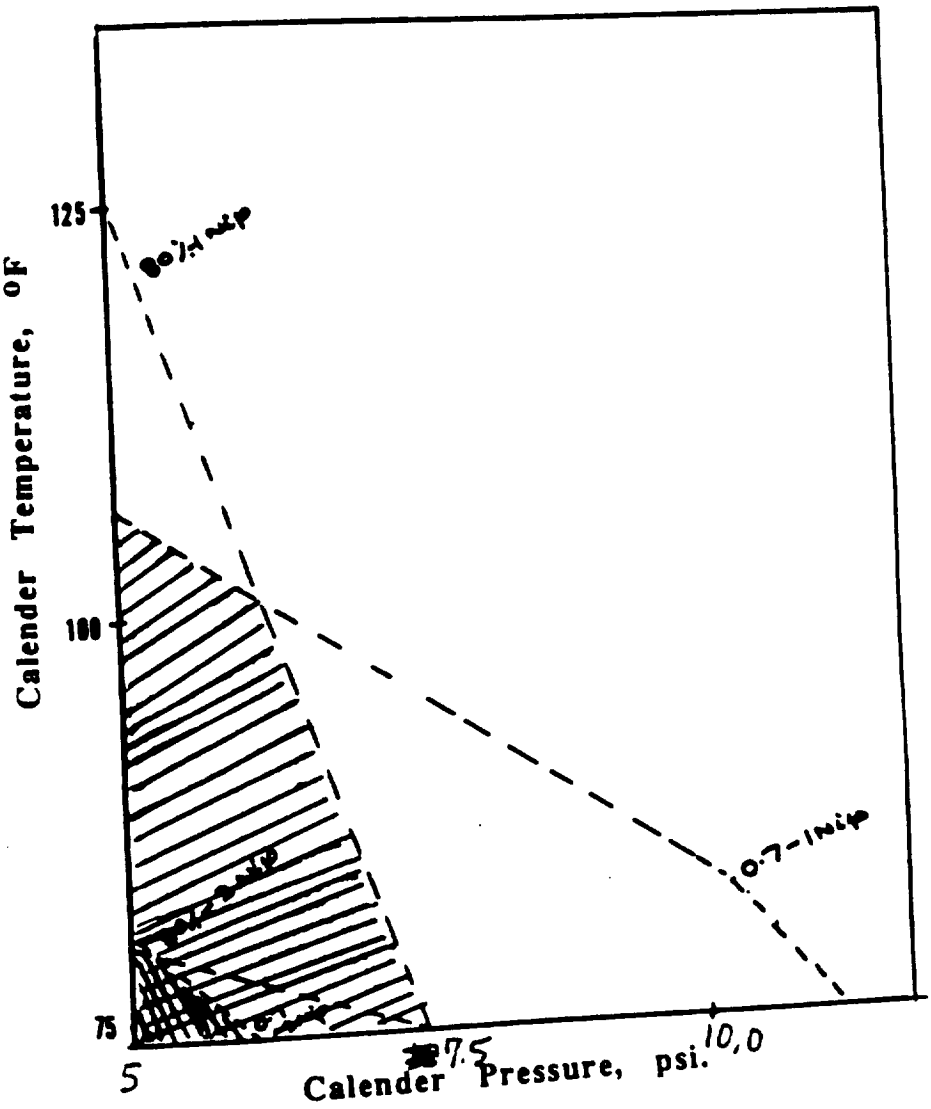


Figure 11

Determination of Required Calendering Conditions
Batch II: 100% PE (51 gsm) + 60% PKT/40% PE (89 gsm)

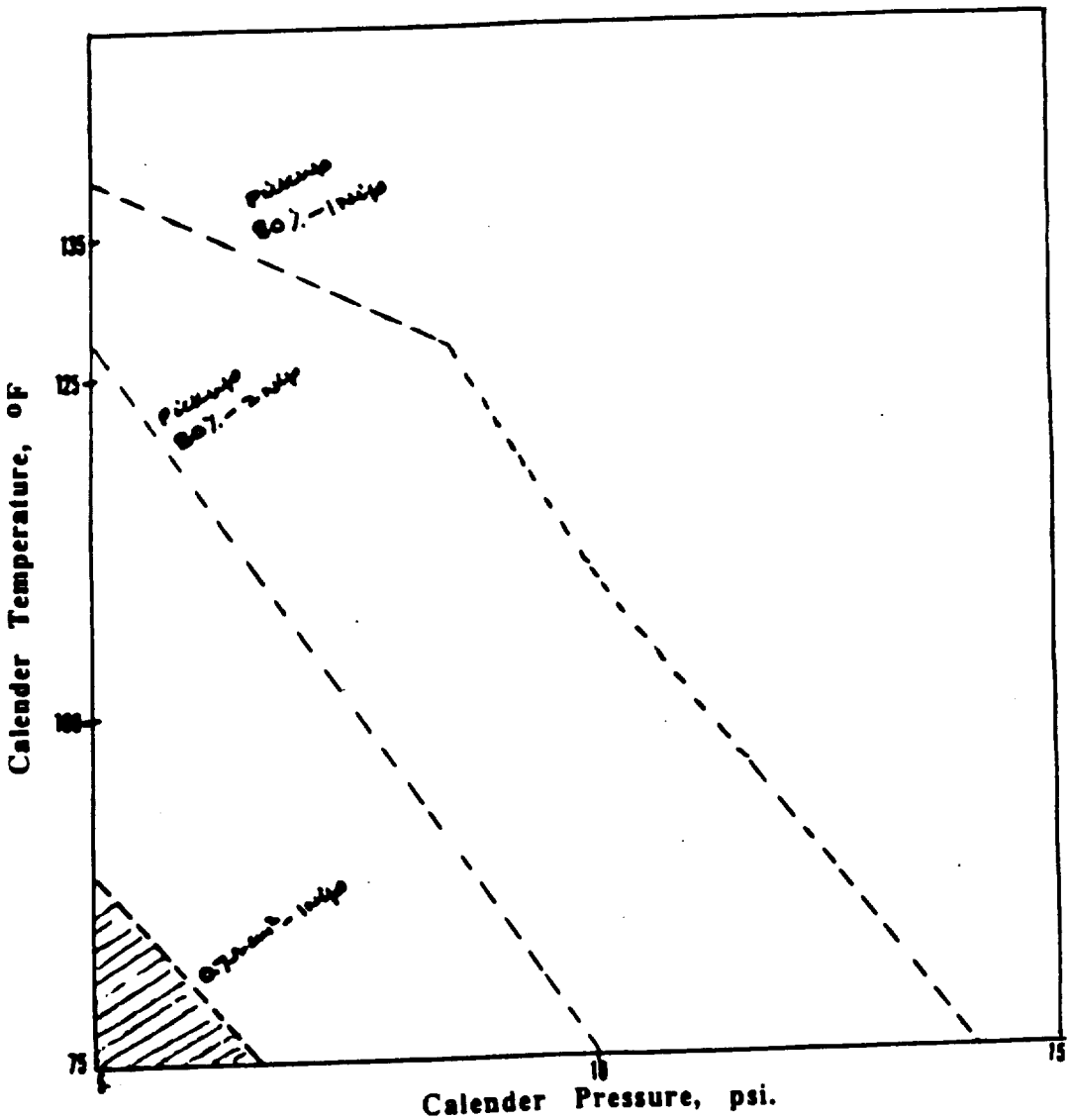


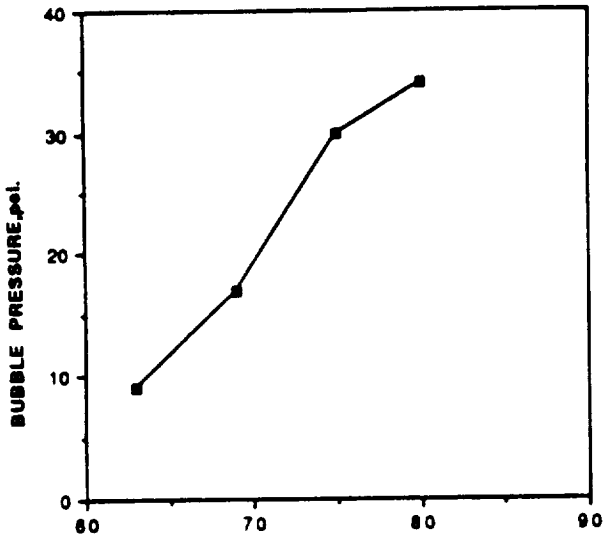
Figure 12

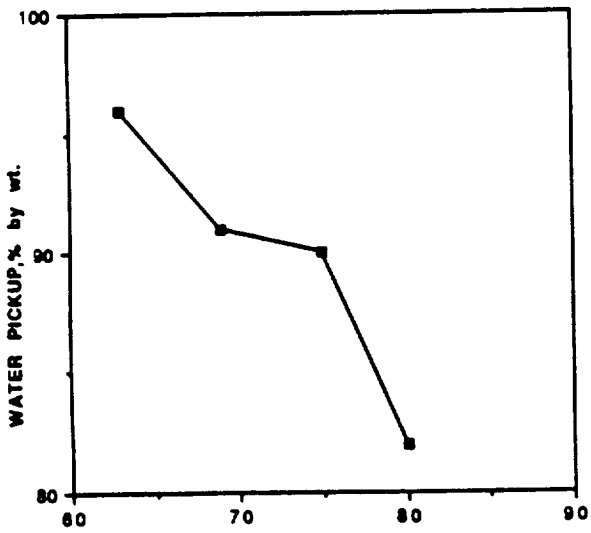
Determination of Required Calendering Conditions
Batch III: 100% PE (51 gsm) + 60% PKT/40% PE (105 gsm)

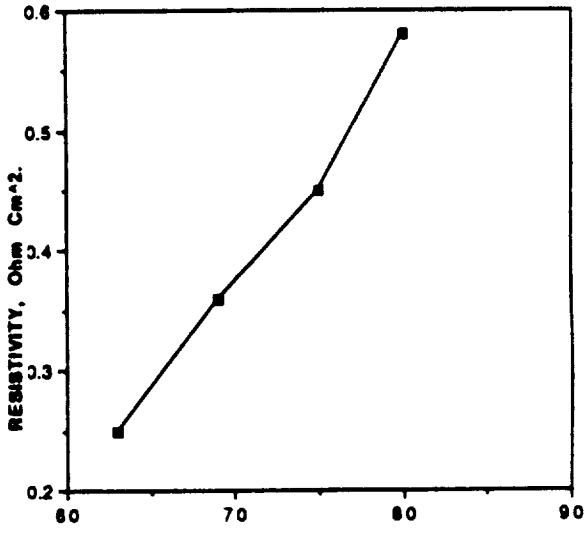
Table IX
Caledering Conditions and Properties of Two Ply Separators BatchII/1 and Batch II/2

TWO PLY SEPARATOR SAMPLES	CALENDER PRESSURE PSI	CALENDER TEMP F	BUBBLE PRESSURE PSI	H2O PICKUP % BY WT.	RESISTIVITY OHM CM^2
BATCH I					
1 NIP	5	RTP	9	96	.25
2 NIP	5	"	12	88	.49
1 NIP	10	"	13	81	.47
2 NIP	10	"	17	68	.84
1 NIP	15	"	16	67	.74
2NIP	15	"	19	66	1.10
1 NIP	20	"	20	60	1.14
2 NIP	20	"	22	57	1.21
BATCH II/1					
1 NIP	5	RTP	17	91	.36
2 NIP	5	"	23	76	.55
1 NIP	10	"	25	75	.51
2 NIP	10	"	24	69	.82
1 NIP	15	"	27	67	.75
2 NIP	15	"	29	63	1.21
1 NIP	20	"	30	63	1.23
2 NIP	20	"	30+	60	1.35
BATCH II/2					
1 NIP	5	RTP	30	90	.45
2 NIP	5	"	30+	80	.57
1 NIP	10	"	30	77	.62
2 NIP	10	"	30+	72	.87
1 NIP	15	"	"	68	.95
2 NIP	15	"	"	66	1.10
1 NIP	20	"	"	68	1.15
2 NIP	20	"	"	64	1.23
BATCH II					
1 NIP	5	RTP	30+	82	.58
2 NIP	5	"	"	81	.67
1 NIP	10	"	"	79	.65
2 NIP	10	"	"	79	.81
1 NIP	15	"	"	75	.87
2 NIP	15	"	"	68	.86
1 NIP	20	"	"	72	.91
2 NIP	20	"	"	66	.99
RTP- Room Temp.		BATCH I	60/40(69 GSM)	+100%PE(51g)	
		BATCH II/1	60/40(69 GSM)	"	
		BATCH II/2	60/40(75 GSM)	"	
		BATCH II	60/40(80 GSM)	"	

Figure 13 Basis Weight of 60%PKT/40%PE Component of Two Ply Separator Vs Properties of Separators







Conclusions

Single Ply Separators

It was concluded that it was not possible to arrive at a satisfactory separator that meets all the given specifications neither with a 100% polyethylene sheet nor with a 60% PKT/40% PE sheet.

Two Ply Separators

It was concluded that a satisfactory separator can be produced when a 100% PE(51 gsm) is bonded together with a 60% PKT/40% PE sheet at grammages greater than or equal to 75 gsm. The required calendering conditions are at room temperature and 5 psi calendering pressure passing through one nip.

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2. Gonzalez-Sanabria, O.D. and Manzo, M. A. "Separator Development and Testing of Ni-H2 cells." Paper presented at 19th Intersociety Energy Conversion Engineering Conference, San Francisco, August 19-24, 1984. Pages 1 - 6. NASA Technical Memorandum 83653.

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